ENG105 Fundamentals of Engineering Lab Practices

Proppant Analysis

frac fluids

Water Analysis

Acidizing

EDC, Tomball

Version 1.25

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Student Manual
ENG105 Fundamentals Engineering Lab Practices
Version 1.25

ENG105 Fundamentals of Engineering Lab Practices

Version 1.25

Printed: 4/26/2007

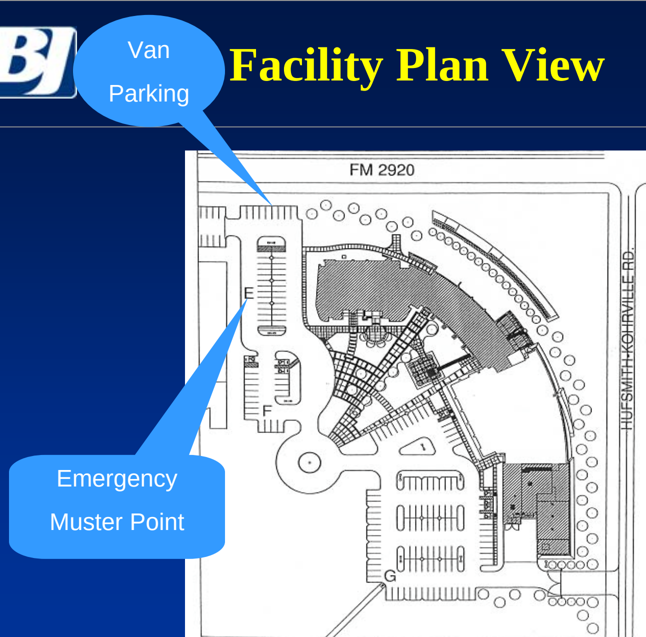
Emergency Procedures

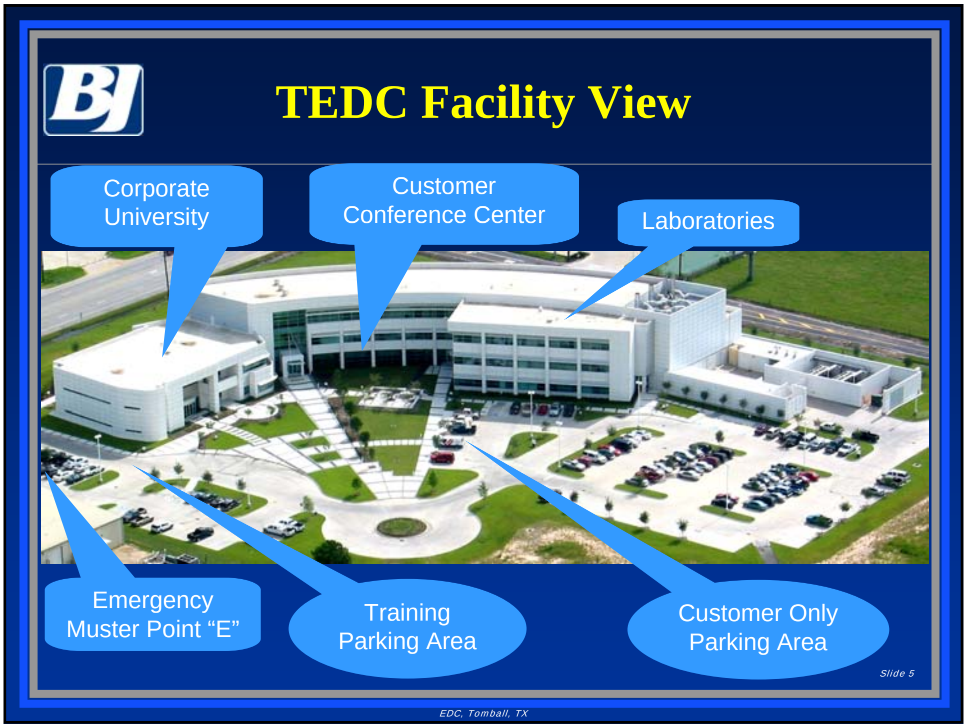
- Evacuation Procedure

Emergency Procedures (cont.)

- General Safety Issues
  - 10 mph Speed Limit in BJ Tomball Parking Lot
  - We have a traffic circle, those in the circle have the right of way!
  - Houston is the 4th largest city in the US
  - Hotel is safe, however!
  - Tobacco Usage Not Allowed in Buildings
    - Smoking
    - Chewing

Facility Plan View




TEDC Facility View
Corporate University
Customer Conference Center
Laboratories
Emergency Muster Point “E”
Training Parking Area
Customer Only Parking Area
Slide 5
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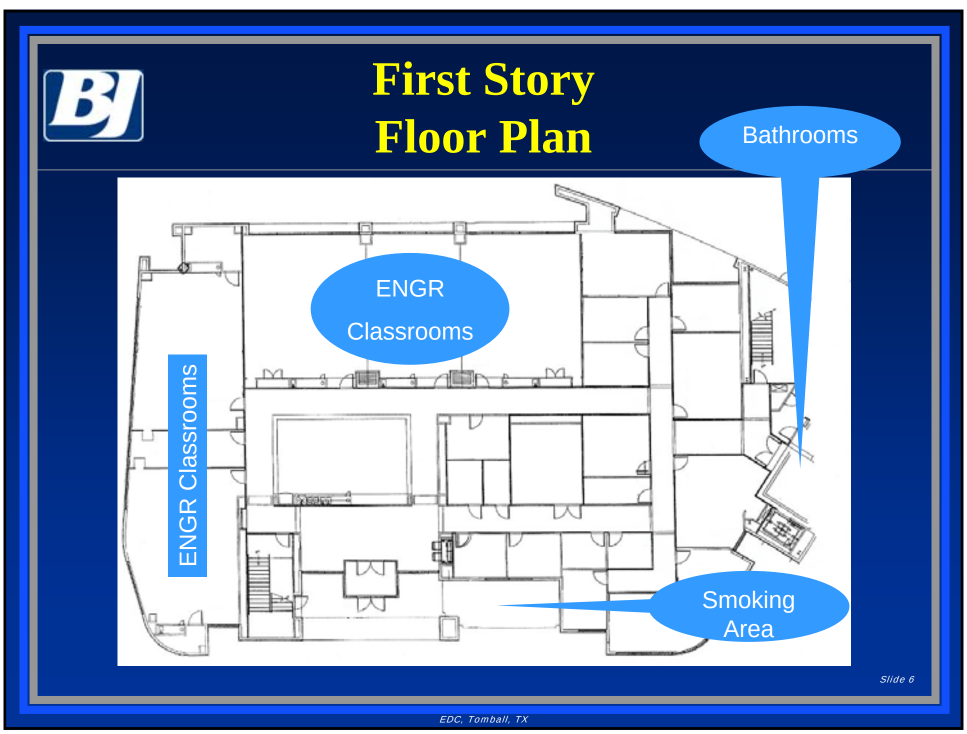
First Story Floor Plan
Bathrooms
ENGR Classrooms
ENGR Classrooms
Smoking Area
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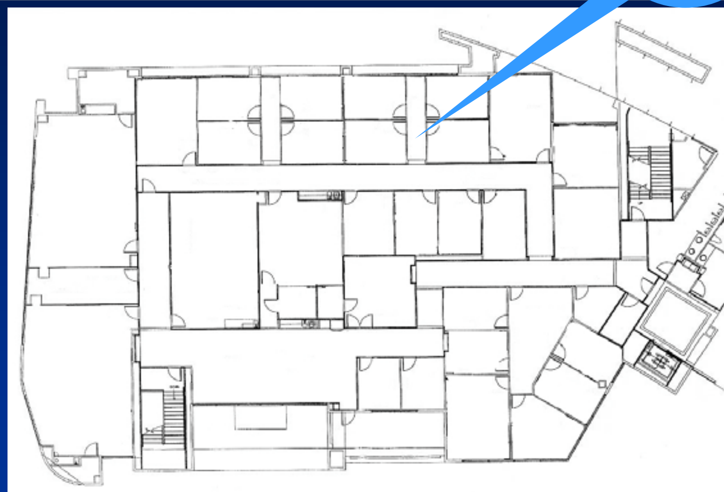
EDC, Tomball, TX

Second Story Floor Plan



Welcome!

- Name Tags MUST be worn at all times
- Approved Areas
  - Employee Development Center
    - The security code for the door is _____. Press the "Start" button, enter the security code, and then press the "Enter" button (white button).
  - Bathrooms
  - Smoking Area
  - Appointments required anywhere else

Welcome! (cont.)

- Standard Classroom Course Procedures
  - Dress Code - Business Casual
    - No Hats in Classroom
    - No Cut-Off Sleeves
    - Shirts with Collars
    - No Sandals
  - Cell Phones, Pagers, Beepers
  - Break Time Intervals
  - Sign-In Procedures
  - Classroom Computer & Laptop Use

Welcome! (cont.)

- General Course Information
  - Course will start promptly at 8:30 AM
  - Please return from lunch and breaks punctually
  - Finishing time about 5:00 PM

Lunches

- We will be eating in the Common Lunchroom
- 1st floor meal time is noon (12:00 hrs)
- Clear tables for next group when finished eating
- Drinks are tea & water (if you want soda - take it in from the Training Dept machine)

- Remember to wear your name tag!

A Message From Executive Management

- During the time you are in a training session, that training is your job.
- You are expected to perform there as you would for your most important customer.
  - You should not leave the classroom to conduct business other than the training class you are in.
  - You should participate fully in the training
    - Therefore, you should turn off your beepers before going to class and leave your cellular phones in your car, room, or other safe place.

A Message From Executive Management (cont.)

- Continued:
  - The training department will give you messages as received, but will not let you out of class to handle these messages, unless there is a bona fide emergency.
  - All messages should be handled during your scheduled breaks.
  - Being late to class, late from breaks, or leaving early is unacceptable behavior.

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A Message From Executive Management (cont.)

- Continued:
  - Classes must be attended in total.
  - You cannot miss a day or part of a day.
    - If you do, you must reschedule that class.
  - You will not receive credit or partial credit for attendance to less than the full curriculum.

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A Message From Training Management

- Remember you are representing BJ even when you are away from the BJ campus.
- Please conduct yourselves as ladies and gentlemen at all times during your stay in Tomball.
- The employee rules governing sexual harassment and hostile work environment will apply during your stay at your hotel.
- BJ Services is paying for your hotel stay and you are representing BJ Services during your stay here.

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A Message From Training Management (cont.)

- Any violation of company rules during your stay at the hotel reported by the hotel staff, will be reported to both the HR department and your District Manager.
- Disciplinary actions will be taken against those violating these policies.
- Please do not disconnect the "Drive-Right" monitors in the training vans.

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ENG105 Fundamentals of Engineering Lab Practices: Course Outline

- Water Analysis
- Proppant Analysis
- Geologist Characterization
- Geomechanical Laboratory
- Acid Laboratory
- Frac Fluid Testing
  - Plus, Equipment Calibration
- Cement Slurry Testing
  - Plus, Lab Equipment Description

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Testing & Grading

- Pretest
  - Test of Current Knowledge – DO NOT GUESS!
- 5 Daily Tests (each = 20% of Final Score)
- Final Score must be 80% or greater

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Class Introductions

- Please introduce yourselves
  - Name
  - Length of service with BJ Services
  - Short career history
  - Place presently assigned
  - Your job title and time in present BJ position
  - Your job responsibilities
  - Knowledge of Oilfield Laboratories

- *Pretest is next – Do Not Guess at Answers!*

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Tomball Technology Center Overview

Section 2

Printed: 4/26/2007

Support Departments

Purchasing	Manufacturing	Software Applications
Warehousing	Laboratory Services	Mechanical Engineering
Training	Technology	Instrumentation
HSE	Water Management	Quality Systems

Tomball Technology Center
TTC

Technology Laboratory Services

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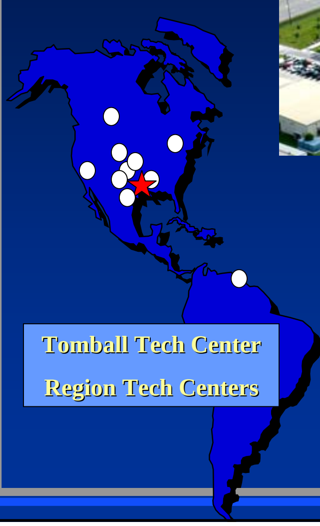
Technical Support



>250 Pumping Service Patents

- Tech Center
  - ISO 9001
  - 51 Acres
  - 360 Employees
  - Laboratories (19) - 36,720 ft^2
  - Manufacturing (20) - 75,000 ft^2
  - Engineering - 72,000 ft^2
  - Warehouse - 131,400 ft^2

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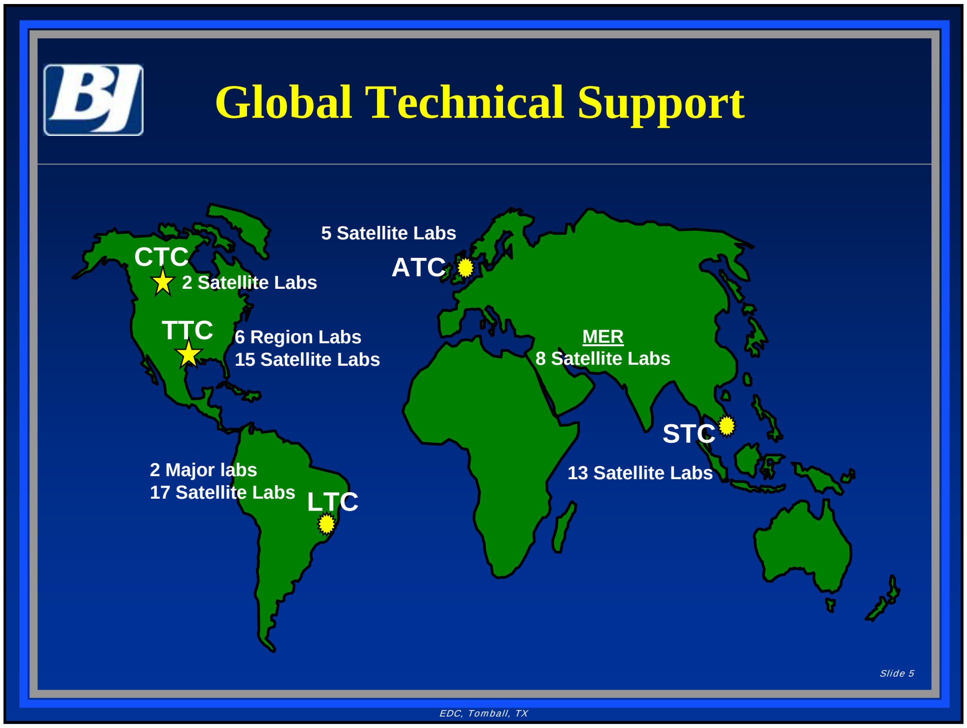
Global Technical Support
5 Satellite Labs
CTC
2 Satellite Labs
ATC
TTC
6 Region Labs
15 Satellite Labs
MER
8 Satellite Labs
STC
2 Major labs
17 Satellite Labs
LTC
13 Satellite Labs
Slide 5
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Water Analysis

Introduction

- **WATER ANALYSIS - Why needed ?**
  - Fracturing fluid - delay or prevent polymer hydration/ X-linking
  - Water is use to gel system, X-linked gel system - fracturing job
- **WATER ANALYSIS - What are we looking for?**
  - CHARACTERIZATION = Quantitative analysis
    - specific gravity, pH
    - cations - K^{+}, Ca^{2+}, Mg^{2+}, Ba^{2+}, Fe^{2+}, and Fe^{3+}
    - anions - Cl^{-}, HCO_3^{-}, SO_4^{-2} , CO_3^{-2},
    - bacteria
- **PROCEDURES**
    - Easy to follow, fast, accurate
- **CALCULATION**

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Sampling Procedure

- Good sampling method - practice
  - **Representative of the system**
    - Most important - the starting point of meaningful results
  - **Water sample**
    - Containers should be **clean or new**
    - 500 ml-Plastic bottles with caps - rinse with sample
    - **Label clearly - proper identification**
  - **Oil/organic sample**
    - Containers should be **clean or new**
    - Glass bottles with proper caps - NO METAL CONTAINER OR CAP
    - **Label clearly - proper identification**
  - **Sample size**
    - On-location - about 200 mls
    - Out side laboratory - 500 mls

Sampling Procedure (con't)

- **Producing well**
  - Wellhead - NOT heater treater or storage tank
- **Fracturing Tank sample**
  - center of tank using thief
  - reaching through the tank top - scooping out a sample
  - bottom valve of the tank - flush out he valve - open valve fully
  - water tested before - blender circulation - prevent contamination
- **Sample Bottle - Storage**
  - bottle - rinse with water samples
  - Tightly cap bottle & labeled properly w/permanent ink
- **Sample identification**

Sampling Procedure (con't)

- **Objective of the analysis**
  - Know exactly what the customer want.
  - Ask lots of questions
  - For Bacteria - Immediate analyses
- **SUMMARY**
  - **Quality of results depend on these three things:**
    - **Representative of the system**
    - **Quality of Sample**
    - **Appropriate Timing**
- **Quality of samples = Quality of results**

Properties

- **pH**
  - A measure of the acidity or alkalinity of water, 0 to 14
  - log$^-$ of the H^+ ion concentration in moles per liter.
  - Most field samples - pH 3.75 to 8.5
  - Drilling fluids are normally alkaline - above pH 8
  - Formation water - in the range of pH 6
  - Presence of unspent acid - pH will be between 1.0 and 3.75
  - Fracturing fluid - pH 6.5 - 8.0 is acceptable
  - pH<6.5 or pH>8.0 - indicates contamination - extra buffering
  - Cement fluid - pH 6.0 - 8.0 is acceptable
  - pH<6 - retard setting time, pH>8 - accelerate setting time
  - Completion fluid - pH< 7.5 is acceptable
  - pH>7.5 - may cause problem
- **Methods**
  - Colorimetric - indicator paper, Potentiometric- pH meter

Properties (Con't)

- pH Testing Procedure
  - pH meter is precise - direct readout of pH over a range of 0 to 14
  - pH meter need to be calibrated using buffer solutions
- Reagents and Equipment
  - 250 ml Beaker
  - Hydrion pH indicator paper
  - pH meter with electrodes
  - Standard pH buffer solutions (pH 4 and pH 7)
- A. pH measurement with Indicator Paper
  - Place the paper into the sample. Leave the paper in the water for about a few seconds. Compare the change in color or color intensity developed on the paper to the pH color chart on the paper container.
- B. pH measurement with a Meter
  - Rinse the pH electrode with distill water and place in the water sample. Set the function switch to the pH position. Read the pH value of the sample.

Properties

- **Specific Gravity**
  - Ratio of the wt of any volume of a substance to the wt of an equal volume of water.
  - Approximate dissolved solids and/or the chloride content.
    - Salt water > pure water
  - Quick check to see if KCl water is being used.
  - 1% KCl - 1.005 @ 60F
  - 2% KCl - 1.011 @ 60F
  - 3% KCl - 1.024 @ 60F

- **Calculation:**
  - specific gravity = $\dfrac{\text{Wt of sample (g)}}{\text{Volume of sample (ml)}}$

Properties (Con't)

- **SPECIFIC GRAVITY - Testing Procedure**
- **EQUIPMENT:**
  - Graduated cylinder (250 ml), Hydrometer (Range of 1.000-1.225)
  - Thermometer (Range of 0° to 230°F), Filter paper (18.5 cm)
  - Filter Funnel
- **Hydrometer Method**
  - Fill a graduated cylinder with the water sample (filtered) to be tested.
  - Place a clean, dry hydrometer in the water. The hydrometer should float freely, not touching the sides or the bottom of the graduated cylinder.
  - Direct reading - hydrometer to the nearest 0.001 **(AT EYE LEVEL)**
  - Take the temperature of the water sample
  - The specific gravity is corrected to 60°F, as follows:
    - For each 5 degrees **above 60°F, Add 0.001**
    - For each 5 degrees **below 60°F**, **Subtract 0.001**
- **Examples:**

  Measured SG = 1.015 @ 85°F

  Corrected SG @ 60°F = 1.015 +0.005 = 1.020

  Measure SG = 1.103 @ 45°F

  Corrected SG @ 60°F = 1.103 -0.003 = 1.100

Properties

- **Alkalinity**
  - Compounds present which shift the pH - which way?
  - Frac - Effect the quality of the gel - poor gel quality
  - Cement - shift pH up - accelerate setting time
  - CBrine - importance - tool to select the correct brine
  - Bicarbonate, Carbonate, Hydroxide **(Chief source - natural waters)**
- **Bicarbonate HCO_3^-**
  - Important ion to consider when predicting scaling tendencies.
  - >700 ppm - potential problem - poor gel quality
  - Present when pH 3.75 to 8.2
- **Carbonate and Hydroxide**
  - present when pH above 8.2
- **Methods**
  - **Titrating - pH indicator** **Phenolphthalein and methyl orange indicator**
  - **pH meter**

Properties (Con't)

- **ALKALINITY - Testing Procedure**
  - Titrating the water with a standard acid, using a pH meter to monitor the pH change.
  - Phenolphthalein and methyl orange indicators may be used if no pH meter is available.
- **REAGENTS & EQUIPMENT**
  - Sulfuric acid (0.01 M (0.02N) N H_2SO_4)
  - Phenolphthalein indicator solution
  - Bromphenol blue indicator solution
  - Bromcresol green indicator solution
  - 250 ml beaker
  - 50 ml - buret, buret clamp and support stand
  - pH meter with electrodes

Properties (Con't)

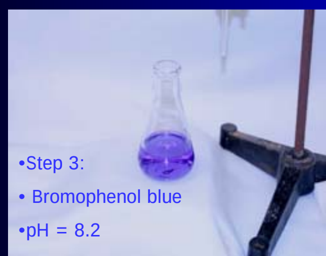
- **pH Indicator - Phenolphthalein endpoint (P)**
  - Measure 50 ml of filtered sample into 250 ml beaker - Record vol.
  - Add 3-drops of **phenolphthalein** to the water sampl
  - **pH > 8.2 = Pink color - If no pink color pH< 8.2 - P = 0**
  - **pH >8.2 - pink -** titrate with 0.01 M sulfuric acid until clear.
  - **pH = 8.2 - Record volume of titrant as P**.
- **pH Indicator - Bromphenol blue**
  - Add 4-drops of **bromphenol blue** indicator - **Blue color pH= 8.2**
  - Titrating until the color changes to a **yellow color** - **pH = 3.75**
  - Record the ***total*** volume of total titrant as amount T.
  - T = phenophthalein + Bromphenol blue

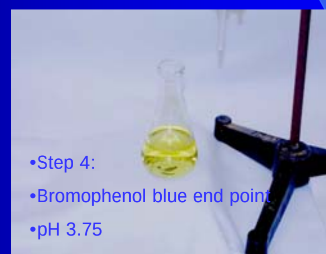
Properties (Con't)

- **pH Meter**
  - Pipette 50 ml of the filtered water sample into a 250 ml beaker. Less than 50 ml may be used if necessary, but the sample size must be recorded for the calculation.
- **Measure the pH of the sample:**
  - If pH >8.2 slowly titrate with 0.01M (0.02N) sulfuric acid until pH reached 8.2
  - Record volume of titrant as P.
  - If pH $\le$ 8.2 then P = 0
  - Continue the titration to **pH of 3.75**
  - Record the ***total*** volume of total titrant (using both indicators) as amount T.

Properties (Con't)

Titration Demo





Properties (Con't)

- **Calculations for Alkalinity Ions**

$$\text{Concentration (mg/L)} = \frac{VF * 1000 * MEF}{\text{Sample size}}$$

VF = volume factor
MEF = miligram equivalent factor

The MEF for each ion is:

Hydroxide (OH^-) = 0.34
Carbonate (CO_3^{2-}) = 0.64
Bicarbonate ($HCO_3$0) = 1.22

- Volume Factor (VF) Table:

Results of Titration	Hydroxide	Carbonate	Bicarbonate
1. P = 0*	0	0	T
2. P < 1/2T	0	2P	T-2P
3. P = 1/2T	0	2P	0
4. P > 1/2T	2P-T	2(T-P)	0
5. P = T	T	0	0

* Representative of most oilfield samples

Properties

- **Chloride**
  - Practically in all oilfield waters - dilute to saturated conc.
    - Most ask analysis - valuable data
    - NaCl, CaCl, MgCl, KCl - chloride analysis
  - High chloride content stimulates the corrosive atmosphere.
  - Frac - >7.0% KCl - potential problem - rheology testing req.
  - CBrine- indication for selecting the correct brine
  - Acid -May cause problem in the acid - HF - System
  - Cement - Cl<10,000ppm is acceptable
  - Fresh - Cl>120,000 - Accelerate; Cl >200,000 - Retard
  - Sea water - different requirement
- **Method (3-4)**
    - Titration -- Silver Nitrate and Mercuric Nitrate
    - HACH kit
    - Ion Chromatography

Testing Procedure - Chloride

- **REAGENTS:**
  - Titrant - 0.0282N Silver Nitrate Solution/ .282N Mercuric Nitrate Solution
  - Chloride Indicator - Potassium Chromate or Chloride 2 Diphenylcarbazone
- **EQUIPMENT:**
  - 1 ml Pipettor, 125 ml Erlenmeyer flask, 125mlBuret
- **PROCEDURE:**
  - **Chloride Titration using Mercuric Nitrate**
    - Sample size into 125 ml flask, dilute to 50 ml w/ DI water
    - Add one **diphenlcarbazone** powder pillow while stirring - **yellow color should develops**
    - Titrate with mercuric nitrate - until **pink color endpoint** .
    - Record mount titrant used as T(Total titrant)

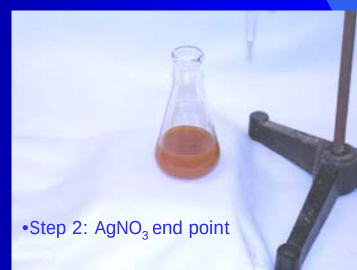
– Chloride Titration Using Silver Nitrate

Pipette specified amount into 125 ml flask, dilute w/ 20 ml of DI water

Specific Gravity	Sample Size
1.000 - 1.015	25 - 50 ml
1.015 - 1.100	3 ml
1.100 - 1.200	1 ml

- Add 1- 2 drops of potassium chromate- **yellow color develops**
- Titrate with 0.0282N silver nitrate - **slight reddish brown**
- **Record titrant as T (total titrant)**
- Sample size should be increased to improve accuracy if the titrant volume is less than 5 ml.

•Step 1: potassium chromate



•Step 2: $AgNO_3$ end point

- **CALCULATIONS: Chloride ion**
  - **Silver Nitrate**
    - Chloride mg/L = $\frac{\text{Vol of titrating solution * 1000}}{\text{mls of Sample Used}}$
  - **Mercuric Nitrate**
    - Chloride mg/L = $\frac{\text{T * 1000 * MEF}}{\text{mls of Sample Used}}$

Note: MEF
0.282 N mercuric nitrate = 0.141 Mol
MEF = 2 * mol mercuric nit * MW Cl
MEF = 2 * 0.141 * 35.45 = 10

- **TECHNICAL NOTES:**
  - The Factor is mg of chloride titrate by 1 ml of 0.0282 N silver nitrate. Normality differs from 0.0282N $AgNO_3$, factor must be changed by the ratio of actual normality to 0.0282N.
  - Example: (Actual / 0.0282) X Factor = (0.282 / 0.0282) X 1000 = **10,000** is the Cl factor for 0.282N $AgNO_3$ would then be 10,000
  - Strips are also available 0-3000 ppm

Properties

- **Calcium and Magnesium**
  - Closely related in water problem & analytical procedure
  - Both Referred to as Total Hardness of water
  - Frac sytem >400 ppm - complex with borate system
  - Acid system -potential problem in - HF system
  - Completion brine - could cause precipitation/brine type
  - Help to determine the source of the water
    - **Calcium**
      - constituents of scale - ($CaSO_4$) or ($CaCO_3$)
      - present in large quantities in Limestones
    - **Magnesium**
      - soluble salt ($MgSO_4$) and ($MgCl_2$)
      - present in large quantities in (dolomite)
- **Method**
  - Titration, total hardness strips, ICP and DCP

- **Testing Procedure - Calcium and Magnesium**
- **EDTA - Ethylenediaminetetraacetic acid** and its sodium salts form chelated soluble complexes when added to solutions of certain metal cations.
- **REAGENTS:**
  - 0.01M (0.02N) EDTA Solution
  - Calver Buffer solution (4M NaOH)
  - Hardness Buffer solution
  - Calver 2 indicator
  - Magnesium CDTA salt powder pillows
  - ManVer 2 indicator
- **EQUIPMENT:**
  - Pipettes (1, 5, 10 and 25)
  - Beakers (250 ml)
  - Buret (25 or 50 ml)

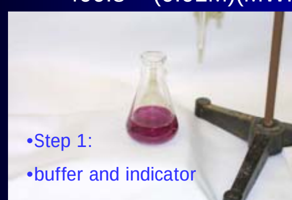
PROCEDURE: Calcium Determination

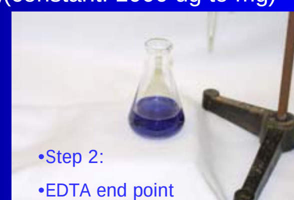
- Pipette 10 ml of sample into a beaker and dilute to 50 ml with distilled water.
- Slowly add 1 ml of CalVer Buffer, vigorously swirling the flask .
- Add Calver Buffer until pH is 12 or higher
- Add CalVer 2 Calcium pillows Indicator - **red-rose color**.
- Titrate with 0.01M (0.02N) EDTA solution until endpoint - **blue**
- Record the volume of EDTA solution titrated as **T1**

Calculation:

- Calcium (mg/L) = $\frac{\mathbf{T1}\ (ml)\ *\ 400.8}{\text{Sample Size (ml)}}$

400.8 = (0.01M)(MW: 40.08)(constant: 1000 ug to mg)


•Step 1:
•buffer and indicator

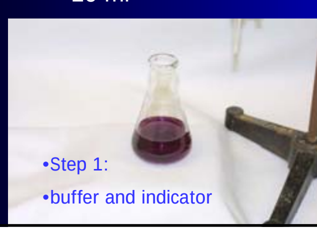

•Step 2:
•EDTA end point

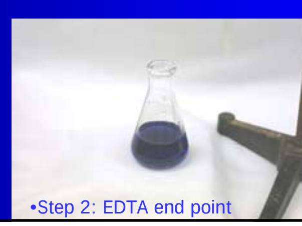
PROCEDURE: Magnesium Determination

- Pipette 10 mls of sample and dilute to 50 ml with distilled water.
- Add 1 powder pillow of Magnesium CDTA.
- Add 5 ml Hardness Buffer to a pH of 10
- Add 1 powder pillow of Manver 2 Indicator - red color
- Titrate with 0.01M EDTA solution until the color turns **blue**
- Record the amount of EDTA titrant used and record as **T2**

- Magnesium (mg/l) = (**T2 - T1**) X 24.3 (24.3 is a factor)

Sample Size	Factor
1 ml	243
10 ml	24.3


•Step 1:
•buffer and indicator


•Step 2: EDTA end point

Properties

- **Iron**
  - Total iron = unfiltered , dissolved iron = filtered
  - Occurs in many formation water - less than 100 ppm
  - Come from Corrosion by-product - Rust, iron sulfide, iron carbonate from acid treatment - can form sludge in oil
  - High iron content will normally be yellow in color
  - Ferric - +3 ppt @ about 2.5, Ferrous - soluble up pH 7.5
  - Acid system - entering the formation system - iron > 10,000 ppm
  - Ferric(+3) = Total - Ferrous (Fe+2 Phenanthroline)
  - Total = TitraVer Titration(10-1000mg/L)
  - Monitor in the completion Brine - corroding problem
- **Method**
  - HACH
  - Inductively Coupled Plasma & Direct Current Plasma

Properties

- Frac -soluble Fe 10 - 25ppm-can harm the gel system
- Acid - raw acid - allowed 100 ppm
- Cement - account for in the pilot test
- Cbrine - monitor flow back - corrosion potential

- **Fe can act as a Reducing Agent**
  - Ferrous (Fe^{2+}) change the oxidation state of transition metal in the crosslinker - gel will not work properly
  - Ferric (Fe^{3+}) can act as complexing agent - typing up site to prevent proper x-linking
- **Presence of Reducing Agents**
  - Filter approximately 200 ml of water into a clean beaker
  - Add 2-3 drops of KMNO4 to the water
  - Stir (do not shake) and note the resulting color:
    - PINK - no reducing agent
    - CLEAR or BROWN - presence of reducing agent

Properties

- **Potassium**
  - – Marker for fracturing treating fluids
    - Any brine where 2% KCl or higher will contain up to 10,000ppm
    - distinguish between returned fracturing / formation water
  - – Mud acid treatment - importance
  - – Na & K + hydrofluoric acid = Na/K - hexaflorosilicate
  - – Quite insoluble and precipitate as a gelatinous mass.
  - – Help to determine the source of water
  - – Frac - high concentration in KCl - see Cl
  - – Cement - high concentration in KCl - see Cl
  - – Acid - in the form of KCl - problem in HF system
- **Method**
  - Strips are also available in a test kit 0-1000 ppm
  - Inductively Coupled Plasma and Direct Current Plasma

Properties

- **Sulfate**
  - – scale calcium sulfate (gypsum), Barium sulfate, and strontium sulfate
  - – Acid - 100 ppm allowed in raw acid
  - – Cement - 1,500 ppm max - acceptable
  - – Completion Brine- compatibility problem
  - – Fracturing - ppm - problem with Vistar sys
- **Method**
  - Turbidimetric Sulfate @ 450nm
  - Ion Chromatography

Properties

- **Barium**
  - Major source of scale due to water incompatibility
  - Barium sulfate ($BaSO_4$) scale is very difficult to correct
  - Techni-Solve 2000 - BJ Chemical Services
  - Precautions need to be taken to prevent co-mingle of Ba and SO_4.
- **Method**
  - Turbidimetric Barium @ 450nm
  - ICP and DCP

Properties

- **Hydrogen Sulfide**
  - Sulfide is a test for the presence of hydrogen sulfide
  - Rotten eggs smell in sour production
  - Concentration could indicates - corrosion
  - 200 ppm can not smell
  - H2S turn sweet well into sour well - bacteria infestation
  - **Method**
    - **Spot Test for Sulfide**
- **Phosphate**
  - Fracturing - 10ppm delay/prevent gel from x-linking
  - Cement - 10 ppm retard the setting of cement
  - Acid - 10 -100 ppm, xlinked and nonxlinked in raw acid
  - CBrine - Scale tendency Ca-phosphate
  - **Method**
    - Colorimetric phosphate, phosphate test kit (HACH)

Properties

- **Sodium**
  - Seldom used to identify formation waters
  - Other constituents are more significant
  - Most often reported as a calculated value **Not** measured value
    - Difference between sum of anion and sum of cation
    - Use to adjust cation-anion balance in the analysis
    - "catch-all" - include any ion present but not actually determined by analysis
  - High value - check why ??
  - Help to determine the source of water
- **Method**
    - Calculation
    - ICP or DCP

Properties

- **Total Dissolved Solids**
  - T.D.S. is used as a check on specific gravity and resistivity.
  - T.D.S. is very useful in avoiding gross errors in water analysis - Establish Chart
  - Represent the sum of the following ions:

• Chloride	Calcium
• Bicarbonate	Dissolved Iron
• Sulfate	Magnesium
• Sodium	

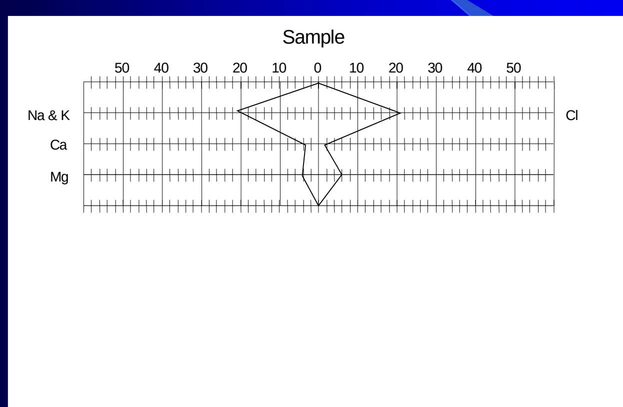
- **Method**
    - Ion summation , Specific gravity

Properties

- Stiff Plot
  - Method to help determine the origin of a water sample
  - Ions found are converted to miliequivalents per liter basis
  - Cations and Anions are plotted on the graph below
  - Pattern act as a fingerprint for a particular water source
    - 2% KCl
    - 15% HCl spent on dolomite
    - 15% HCl spent on limestone
    - formation water………ect.

Properties

- Stiff Plot



BACTERIA

- **What is Bacteria ?**
- Micro-organisms - many different types
  - **Aerobic**
    - Need oxygen to grow
  - **An-aerobic**
    - Grow in the absence of air-or atmospheric oxygen
    - Sulfate reducers
  - **Algae**
    - Contain Chlorophyll - green in color
  - **Fungi**
    - Mold, yeast, grown on living matter
  - **Slime**
    - Condition cause by bacteria or fugus
    - Degrade in the presence of strong acid like guar gums

BACTERIA

- **Problems Caused by Bacteria**
  - **Corrosion**
    - Sulfate reducing bacteria
  - **Formation Plugging**
    - Bacteria growth & bacterial slime
  - **Ferric Hydroxide precipitation**
    - Slime masses
    - Iron bacteria
  - **Fracturing Fluid**
    - Dropped in viscosity in a short period of time
    - Lowering of the pH after the water has set for a few days

BACTERIA

- Bacteria Count - Turner ATP Luminometer
  - Every living organism contains ATP(Adenosine Triphophate)
  - can not distinguish between Algae/Bacteria
  - Salt will interfere with analysis
  - Measure light given off by RXN
  - Luciferin-Luciferase reagent - sensitive to contaminant
- Concentration (bacteria /ml)
  - City water supply - 10 to 100
  - moving surface water - 100 to 100,000
  - Stagnant water - >100,000
- Acceptable Limites
  - 10 to 1,000 use with no biocide
  - 1,000 to 800,000 use with biocide
  - >800,000 dispose of the water
  - Cbrine - Biocide needed - below 9.5 lb/gal

Base Water: Compatibility Range

Properties	Acid	Cement	Frac	C-Brine
pH	15% HCl	6 – 8	6.5 – 8.0	<7.5
Akalinity	Not exist	Not to shift pH>8.0	400 – 700 ppm 500 Vistar	Not to shift PH>7.5
Chloride	HF-system Problem	<10,000pp	>7.0% KCl test needed	NaCl Compatible problem
Total Hardness	HF-system Problem	Pilot Test	400 ppm Max Borate 250ppm Max Vistar	Compatible Problem
Iron	Raw acid 100 ppm Max	Pilot Test	10 – 25 ppm	Monitor – corrode problem
Potassium	KCl See Cl	KCl See – Cl	KCl See – Cl	KCl See Cl

Base Water: Compatibility Range

Properties	Acid	Cement	Frac	C-Brine
pH	15% HCl	6 – 8	6.5 – 8.0	<7.5
Sulfate	Raw acid 100ppm Max	1500ppm Max	200 ppm Max Vistar	Compatible Problem
Phosphate	10 – 100 ppm xlink - nonxlnk	10 ppm Max	10 ppm Max	Scale problem
Sulfide	200 ppm can not smell Dangerous			Zinc, Fe Scale problem
Sodium	NaCl	NaCl	NaCl	NaCl Compatibility
Bacteria		800,000 bacteria/ml Max	800,000 bacteria/ml Max	9.5lb/gal no biocide

ACIDIZING

MEASURED ION	Cross-Linked Acid Systems Max conc.	Specialty Acid Systems Max conc.	Economy Acid Systems Max conc.
Fluoride	10	100	100
Phosphates	10	100	100
Sulfates	600	600	1500
Sulfites	100	100	200
Iron	100	100	125
Arsenic	2	2	2

ACIDIZING

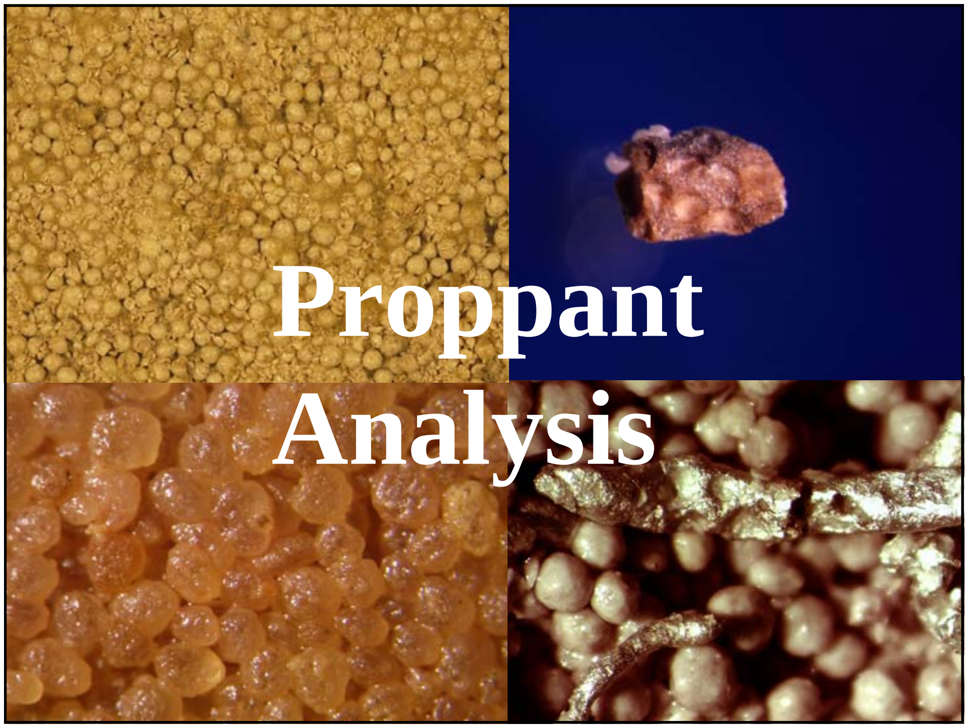
MEASURED ION	Cross-Linked Acid Systems Max conc.	Specialty Acid Systems Max conc.	Economy Acid Systems Max conc.
Total Metals other than Iron & Arsenic (such as Ti, Cu & Pb)	10	10	10
Total Free Halogens	100	100	100
Total Organics	25	25	25

Sample Preparation

- **Water sample mixed with Oil**
  - **Remove oil content**
    - cotton ball in funnel and filtered through
    - Turning the sample over
- **Emulsion with little free water**
  - **Break the emulsion**
    - place sample in separatory funnel and add approx. 10 drops of non-emulsifying agent/surfactant
    - shake vigorously - water should break and fall to the bottom
    - drain water sample out into a funnel with cotton
    - Note: some time you may have to heat the sample before adding NE- agent or surfactant

Sample Preparation

- **Water with high Iron contents**
  - **ppt out the Fe**
    - Take 5-mls of sample and add 10mls of 4M NaOH - result with a thick precipitation
    - Filter off the solid and wash with 5-ml of DI water - RETAIN THE WATER
    - check the pH of the water portion, add a few drops of 15% HCl to pH $\leq$ 4
    - dilute to 25 ml and treat the sample by normal method
    - Remember: At this point the dilution factor is five (5).
- **Water with high Sulfides contents**
  - **Remove sulfide**
    - Add 1-ml of Nitric Acid to 50 ml sample and boil solution slowly for about 15 minutes
    - After boiling, dilute the solution back to 50 ml - readjust pH as necessary

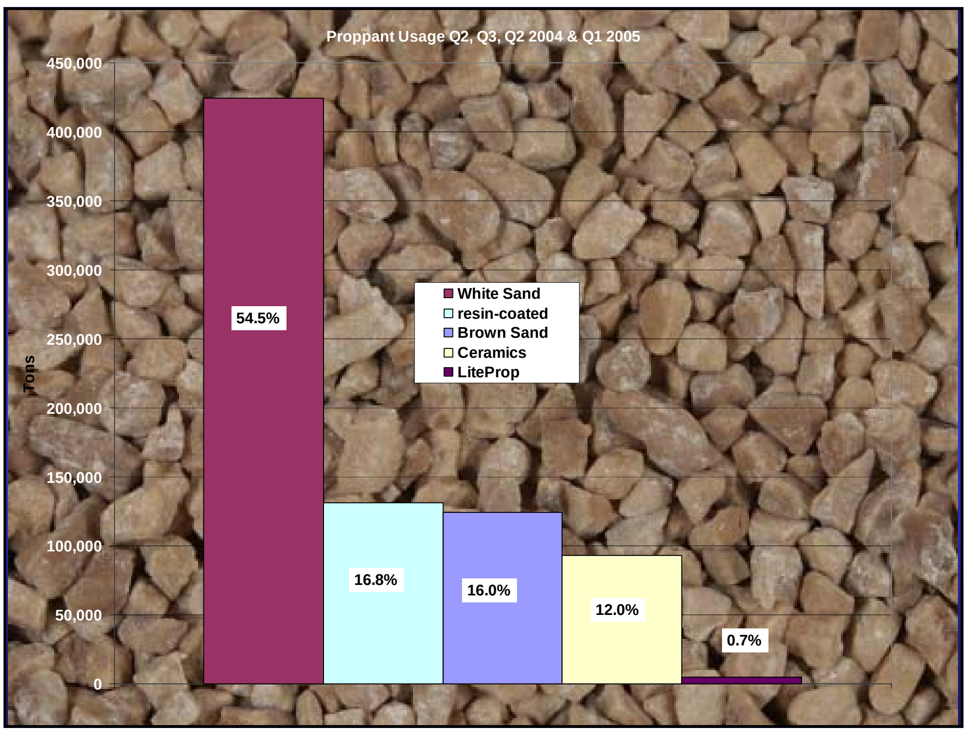


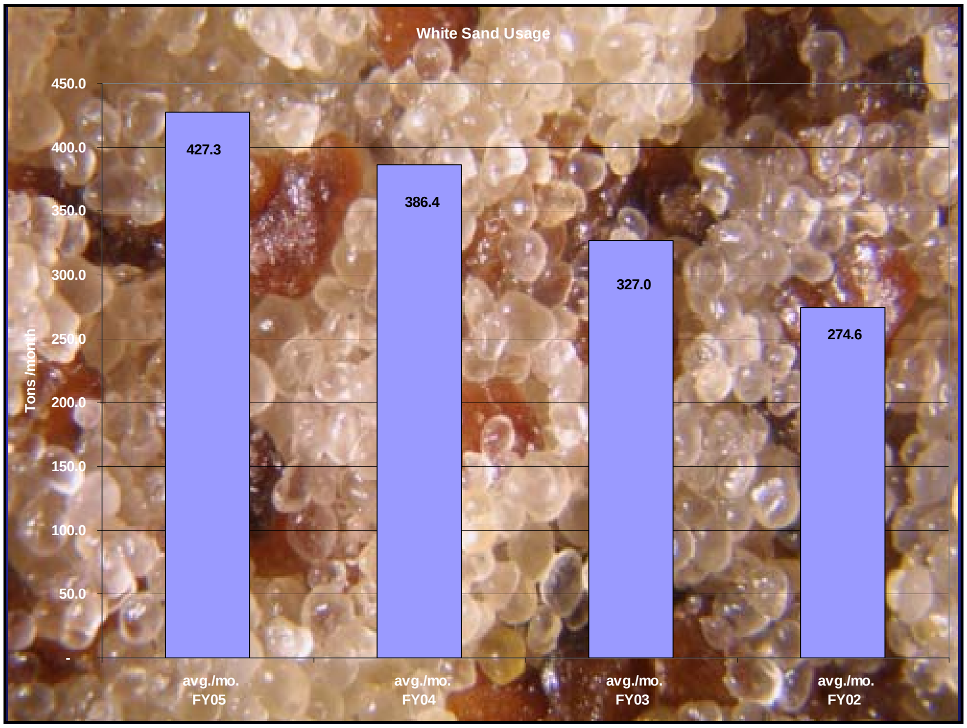
Proppant Analysis

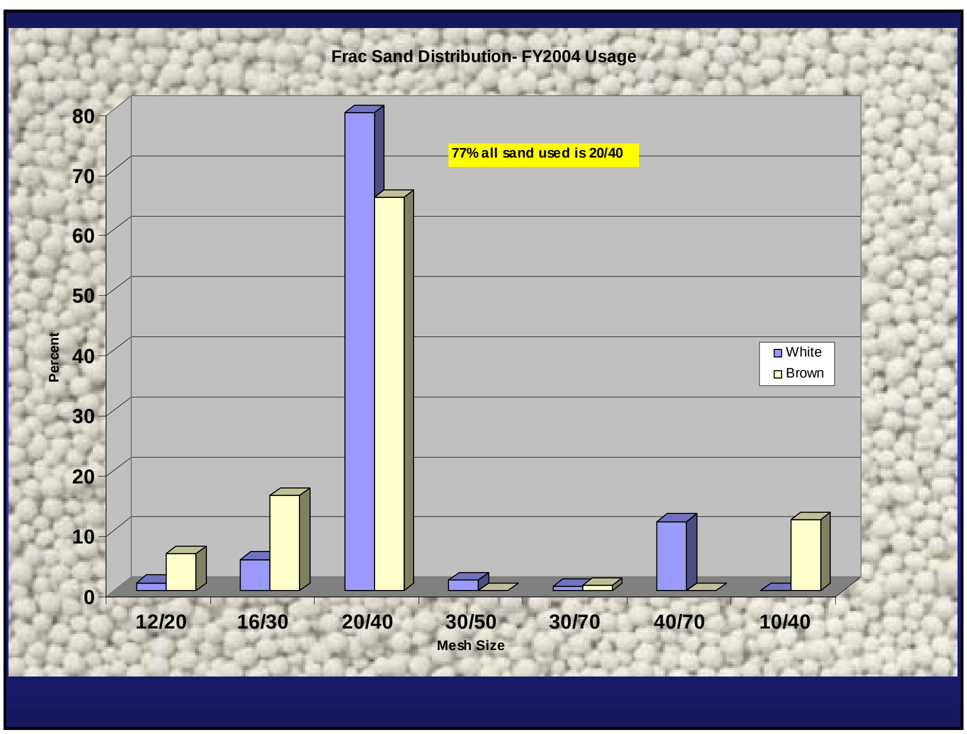
- **API RP 56 -**Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations
- **API RP 60 -**Recommended Practices for Testing High-Strength Proppants Used in Hydraulic Fracturing
- **API RP 58 -**Recommended Practices for Testing Sand Used in Gravel Packing Operations

API Recommended Practice 56

- Second Addition 1995
- To improve the quality of material
- To evaluate the physical properties for comparison.
- To enable users to select materials most useful for application in Hydraulic Fracturing




White Sand Usage
450.0
400.0
350.0
300.0
250.0
200.0
150.0
100.0
50.0
-
Tons /month
427.3
386.4
327.0
274.6
avg./mo. FY05
avg./mo. FY04
avg./mo. FY03
avg./mo. FY02


Frac Sand Distribution- FY2004 Usage
77% all sand used is 20/40
80
70
60
50
40
30
20
10
0
Percent
White
Brown
12/20
16/30
20/40
30/50
30/70
40/70
10/40
Mesh Size

	Conductivity Analysis								
Start Test Date	8/23/1999								
Company	BJ Services								
Representative	Ron Matson								
Eng.Support No.									
Cell #	WHast.	Top							
Width Core Top	0.368	Fluid	0	mls					
Width Core Bottom	0.319	Proppant	63	grams					
Width Pack, initial	0.240								
Fluid	NA								
Additives									
		Proppant	Unimin						
Temperature	150-250	Type	20/40				3,099	50 hour average	
Closure Pressure	1000-6000	Concentration	2	lbs/ft2					
Fluid Pressure	400	Baseline	250@1000psi	Darcies					
Test Data	Temp	Temp	Rate	Viscosity	DP	Width	Conductivity		Closure
Time	°F	°C	mls/min	cp	psi	inches	md-ft	darcies	psi
0	135.94	57.74	11.80	0.48	0.04158	0.222	3,670	198	2033
10	203.69	95.38	7.98	0.30	0.01793	0.222	3,529	191	2026
20	203.61	95.34	7.98	0.30	0.01772	0.222	3,574	193	2023
0	203.65	95.36	7.97	0.30	0.02337	0.215	2,708	151	4025
10	204.04	95.58	7.97	0.30	0.02448	0.215	2,579	144	3995
20	203.54	95.30	7.98	0.30	0.02501	0.215	2,534	141	3984
0	244.13	117.85	9.35	0.24	0.03727	0.207	1,585	92	6073
10	253.64	123.13	7.98	0.22	0.03681	0.207	1,304	76	6066
20	253.75	123.20	7.97	0.22	0.03681	0.207	1,303	76	6052

Sand Sampling

Sampling Device - slot 8 inches x 6 inches x 4 inches

Sample Splitter - Sample reduction
Representative sample

API Requirements
- 9 samples per rail car
- 3 samples per truck
- a minimum of 5 samples per 100,000 lbs.

Sampling

- The sampling device, with it's longitudinal axis perpendicular to the flowing stream, should be passed through the flowing sand stream at a uniform rate.
- Sand should be allowed to flow for at least 2 minutes prior to taking samples
- Samples are combined, split to test size and a single sample is tested

Sample Splitting

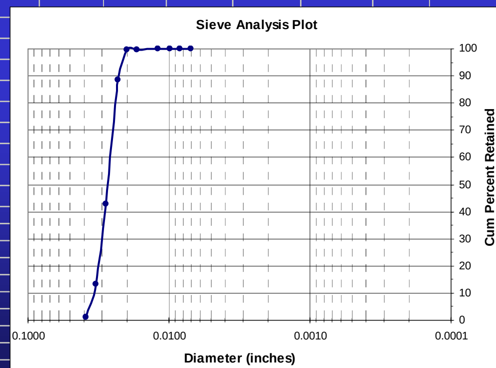
Sieve Analysis

- 6 sieves plus a pan
- Split sample of approximately 100 grams
- Add sample to the top sieve place on a Rotap for 10 minutes
- Calculate wt.% held on each sieve
- The cumulative weight should be within 0.5% of the initial weight.

Sieve Analysis - results

- 90% should fall between designated sieves
- Not over 0.1% should be larger than the largest sieve
- Not more than 1% smaller than the smallest sieve

WELL INFORMATION				**Sieve Size**	**Sieve Opening**	**Empty Weight**	**Final Weight**	**Sand Weight**	**Percent**	**Cum %**
Date:	5/22/03			18	0.0390	402.12	402.88	0.76	1.14%	1.14
Sample No.:	Ottawa-Wedron			20	0.0330	379.84	387.93	8.09	12.17%	13.31
Company:	20/40			25	0.0280	367.46	387.06	19.60	29.47%	42.78
Field:				30	0.0230	361.66	392.08	30.42	45.74%	88.53
Well No.:				35	0.0200	346.93	354.36	7.43	11.17%	99.70
Depth:				40	0.0170	335.60	335.67	0.07	0.11%	99.80
ACID SOLUBILITIES				50	0.0120	330.92	330.98	0.06	0.09%	99.89
15% HCl:				60	0.0098	0.00	0.00	0.00	0.00%	99.89
12:3% HCl:HF:				70	0.0083	0.00	0.00	0.00	0.00%	99.89
				80	0.0070	0.00	0.00	0.00	0.00%	99.89
SIEVE RESULTS				PAN	0.0010	360.09	360.16	0.07	0.11%	100.00
	Inches	μm								
D_{10}:	0.03463	880						66.50		
D_{20}:	0.03186	809						Total Fines	0.20%	
D_{30}:	0.03017	766								
D_{40}:	0.02847	723								
D_{50}:	0.02721	691								
D_{60}:	0.02612	663								
D_{70}:	0.02502	636								
D_{80}:	0.02393	608								
D_{90}:	0.02260	574								
U_C (D_{40}/D_{90}):	1.3	well sorted								
D_{50} :	0.0272	691								
D_{50} x 6:	30 U.S. Mesh									
Gravel Size:	20-40									

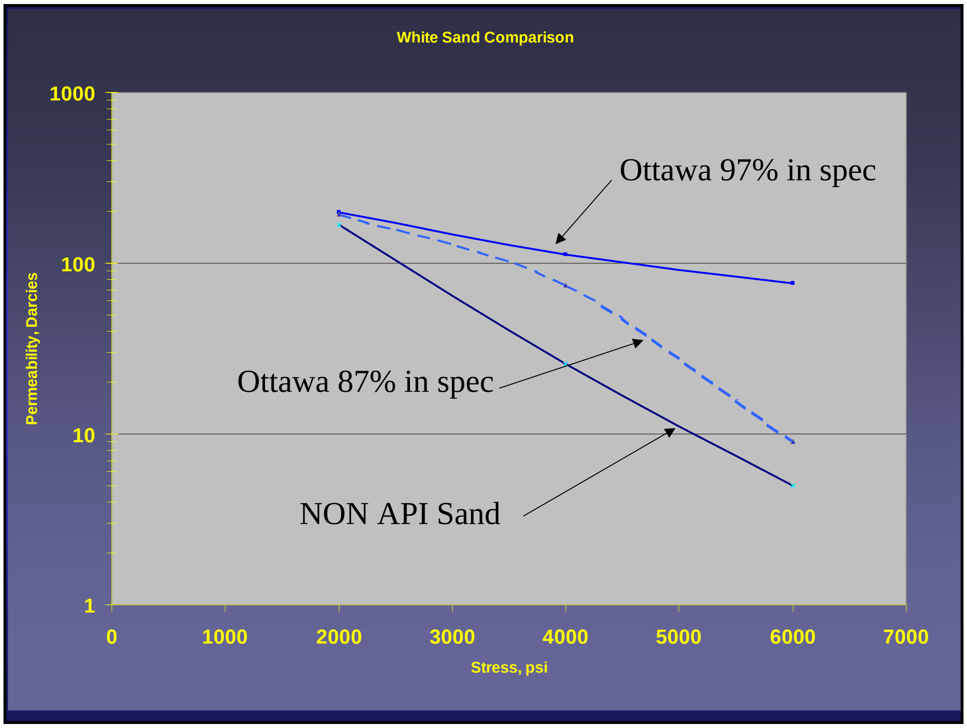


2040 Frac	1979	1993	2001	Present
20	0.5	0.2	0.0	0.0
30	27.5	22.5	17.5	19.8
35	43.7	30.8	44.5	42.6
40	22.8	44.2	30.2	31.0
50	5.2	3.1	7.0	6.2
PAN	0.8	0.6	0.8	0.2
PERCENT MATERIAL	94.0	97.5	92.2	93.2
NTU	135	100	65	43

All results are typical and are based on Badger Mining Corporation's test equipment.

U.S. Sieve No.	20/40 White Sand % retained	20/40 Ceramic HSP % retained
16	0.0	0.0
20	0.5	7.5
30	29.6	90.3
35	33.9	2.0
40	27.4	0.2
50	7.9	0.0
pan	0.8	0.0
TOTAL	100.1	100.0
IN-SIZE	90.9	92.5
Median Diameter	0.538	0.719

U.S. Sieve No.	1979 % retained	1993 % retained	2001 % retained	2003 Q1 % retained
16	0.0	0.0	0.0	0.0
20	0.5	0.2	0.0	0.0
30	27.5	22.5	17.5	19.8
35	43.7	44.2	44.5	42.6
40	22.8	30.8	30.2	31.0
50	5.2	3.1	7.0	6.2
pan	0.8	0.6	0.8	0.2
TOTAL	100.5	101.4	100.0	99.8
IN-SIZE	94.0	97.5	92.2	93.4
Median Diameter	0.546	0.536	0.528	0.531



	Conductivity Analysis							
Start Test Date	6/13/2005							
Company	BP Production							
Representative	Rocky Freeman							
Eng.Support No.	05-05-0580							
Cell #								
FlexSand	0	mls						
Proppant	63	grams						
Width Pack, initial	0.240	inches						
Fluid								
Additives								
		Proppant	Ottawa					
Temperature	150°F	Type	20/40					
Closure Pressure	4500-psi	Concentration	2	lbs/ft2				
Fluid Pressure	500-psi	Baseline	147	Darcies				
Test Data								
Time	Temp	Closure	Conducti	DP	Rate	Viscosity	Width	
Hours	°C	psi	md-ft	psi	mls/min	cp	inches	Darcies
0	65.1	4446	1642	0.0841	11.9220	0.432626	0.215	92
10	65.5	4428	1679	0.0816	11.8929	0.430244	0.213	95
20	65.5	4450	1773	0.0770	11.8439	0.430393	0.213	100
30	65.5	4447	1676	0.0819	11.9122	0.430244	0.213	94
40	65.6	4446	1742	0.0783	11.8468	0.429948	0.213	98
50	65.3	4447	1766	0.0780	11.9077	0.431879	0.213	99

	Conductivity Analysis								
Start Test Date	6/6/2005								
Company	BP Production Company								
Representative	Rocky Freeman								
Eng.Support No.	05-05-0480								
Cell #									
FlexSand	0	grams							
Proppant	63	grams							
Width Pack, initial	0.229	inches							
Fluid									
Additives									
		Proppant	Brady						
Temperature	150°F	Type	20/40						
Closure Pressure	4,500	Concentration	2	lbs/ft2					
Fluid Pressure	500	Baseline	42	Darcies	@4500-psi				
Test Data									
Time		Temp	Closure	Conducti	DP	Rate	Viscosity	Width	Permeability
Date	Hours	°C	psi	md-ft	psi	mls/min	cp	inches	md-ft
6/6/2005	0	64.9	1010.7	10120	0.0237	20.6	0.434	0.228	533
6/6/2005	0	65.0	4498.2	2290	0.0525	10.4	0.433	0.213	129
6/6/2005	10	64.9	4510.1	787	0.0483	3.3	0.434	0.212	45
06/07/05	20	65.0	4507.2	774	0.0495	3.3	0.434	0.211	44
06/07/05	30	65.0	4476.8	774	0.0496	3.3	0.433	0.211	44
06/08/05	40	65.0	4470.2	740	0.0515	3.3	0.434	0.211	42
06/08/05	50	64.8	4475.7	746	0.0513	3.3	0.435	0.211	42

Conductivity Analysis

Start Test Date	6/10/2005								
Company	BP Production Company								
Representative	Rocky Freeman								
Eng.Support No.	05-05-0480								
Cell #									
FlexSand		mls							
Proppant		grams							
Width Pack, initial		inches							
Fluid									
Additives									
		Proppant	Brady with 7% 14/30 FlexSand MSE						
Temperature	150	Type	20/40						
Closure Pressure	4,500	Concentration	2	lbs/ft2					
Fluid Pressure	500	Baseline		Darcies					
Test Data									
Time	Temp	Closure	Conducti	DP	Rate	Viscosity	Width		
Hours	°C	psi	md-ft	psi	mls/min	cp	inches	darcies	
0	64.1	1114	5787	0.0218	10.7	0.438894	0.243	286	
10	66.0	1116	5337	0.0230	10.7	0.427422	0.241	266	
0	64.5	4516	1885	0.0664	10.7	0.436535	0.218	104	
10	64.7	4508	1373	0.0911	10.7	0.43548	0.216	76	
20	64.1	4510	1297	0.0972	10.7	0.439487	0.214	73	
30	65.4	4513	1223	0.1012	10.7	0.430959	0.213	69	
40	65.0	4512	1204	0.1033	10.7	0.433616	0.213	68	
50	66.0	4512	1172	0.1047	10.7	0.427309	0.213	66	

	Conductivity Analysis								
Start Test Date	5/9/03								
Company	BJ Services								
Representative	Matt Kedzierski								
Eng.Support No.	03-04-0339								
Cell #	WHast.	Top							
Width Core Top		Fluid		mls					
Width Core Bottom	10.350	Proppant	63	grams					
Width Pack, initial	9.630	Flex HS		grams					
Fluid									
Additives									
							6,252	50 hour average	
Temperature	150	*Type*	Bakersfield low cost alternative frac sand						
Closure Pressure	1000-5000	*Concentration*	20/40	lbs/ft2					
Fluid Pressure	500	*Baseline*	2	Darcies					
Test Data	Temp	Temp	Rate	Viscosity	DP	Width	Conductivity		Closure
Time	°F	°C	mls/min	cp	psi	inches	md-ft	darcies	psi
0	72.37	22.43	5.32	0.945	0.017	0.222	7,883	426	1,012
10	148.65	64.81	5.54	0.435	0.011	0.222	5,622	304	1,111
20	148.63	64.80	5.44	0.435	0.012	0.222	5,252	284	1,073
30	148.61	64.79	5.24	0.435	0.012	0.222	4,891	264	1,043
40	148.69	64.83	5.35	0.435	0.014	0.221	4,294	233	1,422
50	148.69	64.83	5.35	0.435	0.015	0.221	4,210	229	1,468
0	148.61	64.78	5.54	0.435	0.043	0.215	1,503	84	3,171
10	148.62	64.79	5.57	0.435	0.047	0.214	1,374	77	3,186
20	148.64	64.80	5.57	0.435	0.051	0.214	1,270	71	3,204
30	148.67	64.82	3.00	0.435	0.053	0.214	653	37	3,159
40	148.62	64.79	5.60	0.435	0.056	0.214	1,163	65	3,165
50	148.61	64.78	5.60	0.435	0.056	0.214	1,163	65	3,166
0	148.64	64.80	5.59	0.435	0.143	0.210	455	26	4,979
10	148.66	64.81	5.40	0.435	0.170	0.207	370	21	4,935
20	148.67	64.82	5.43	0.435	0.191	0.206	330	19	4,923
30	148.66	64.81	5.42	0.435	0.191	0.206	330	19	4,922
40	148.66	64.81	5.48	0.435	0.200	0.205	319	19	4,917
50	148.65	64.80	5.51	0.435	0.202	0.209	317	18	4,928

	Conductivity Analysis								
Start Test Date	12/11/02								
Company									
Representative									
Eng.Support No.									
Cell #	WHast.	Top							
Width Core Top	9.350	Fluid		mls					
Width Core Bottom	9.640	Proppant		grams					
Width Pack, initial		Flex HS		grams					
Fluid									
Additives									
		Proppant	Wedron				6,690	50 hour average	
Temperature	150	*Type*	20/40						
Closure Pressure	2000-6000	*Concentration*	2	lbs/ft2					
Fluid Pressure	450	*Baseline*		Darcies					
Test Data	Temp	Temp	Rate	Viscosity	DP	Width	Conductivity		Closure
Time	°F	°C	mls/min	cp	psi	inches	md-ft	darcies	psi
0	99.21	37.34	5.95	0.687	0.01303	0.252	8,397	400	2,013
10	242.73	117.07	3.10	0.238	0.00341	0.222	5,802	314	2,185
20	250.15	121.19	3.16	0.229	0.00330	0.222	5,871	317	2,175
30	250.11	121.17	3.17	0.229	0.00320	0.222	6,070	328	2,168
40	250.05	121.14	3.06	0.229	0.00320	0.221	5,862	318	2,124
50	250.04	121.13	3.64	0.229	0.00388	0.221	5,751	312	2,149
0	248.51	120.28	3.00	0.231	0.00355	0.217	5,214	288	3,105
10	249.05	120.58	3.15	0.230	0.00905	0.215	2,143	120	3,911
20	250.07	121.15	2.76	0.229	0.00924	0.215	1,832	102	3,953
30	250.13	121.18	2.80	0.229	0.00931	0.215	1,842	103	3,980
40	249.33	120.74	2.80	0.230	0.00940	0.215	1,833	102	3,974
50	249.33	120.74	2.80	0.230	0.00939	0.215	1,835	102	3,974
0	250.14	121.19	2.80	0.229	0.01138	0.210	1,508	86	5,599
10	250.08	121.15	2.59	0.229	0.01522	0.209	1,042	60	5,988
20	249.54	120.85	2.56	0.230	0.01583	0.208	995	57	5,965
30	249.73	120.96	2.47	0.229	0.01639	0.207	927	54	5,959
40	249.57	120.87	2.47	0.229	0.01708	0.207	890	52	5,961
50	250.14	121.19	3.00	0.229	0.01785	0.207	1,031	60	5,984

	Conductivity Analysis								
Start Test Date	12/11/02								
Company	BJServices								
Representative	Nathan Anderson								
Eng.Support No.	02-11-0866								
Cell #	WHast.	Top							
Width Core Top	10.300	Fluid		mls					
Width Core Bottom	9.350	Proppant		grams					
Width Pack, initial	0.252								
Fluid									
Additives									
		Proppant	Unifrac SandLot#01661						
Temperature	150	*Type*	20/40				5,391	50 hour average	
Closure Pressure	2000-6000	*Concentration*	2	lbs/ft2					
Fluid Pressure	415	*Baseline*		Darcies					
Test Data	Temp	Temp	Rate	Viscosity	DP	Width	Conductivity		Closure
Time	°F	°C	mls/min	cp	psi	inches	md-ft	darcies	psi
0	98.00	36.67	5.66	0.70	0.0136	0.25	7,759	369	2,013
10	242.71	117.06	2.56	0.24	0.0030	0.22	5,496	297	2,185
20	250.15	121.19	2.48	0.23	0.0031	0.22	4,920	266	2,175
30	250.11	121.17	2.45	0.23	0.0031	0.22	4,897	265	2,170
40	250.06	121.15	2.26	0.23	0.0030	0.22	4,618	251	2,107
50	250.05	121.14	2.46	0.23	0.0032	0.22	4,657	253	2,150
60	249.98	121.10	2.30	0.23	0.0031	0.22	4,573	248	2,097
70	250.16	121.20	2.48	0.23	0.0031	0.22	4,830	262	2,118
0	248.43	120.24	2.76	0.23	0.0042	0.22	4,028	223	3,222
10	249.06	120.59	2.54	0.23	0.0069	0.22	2,288	128	3,918
20	250.08	121.16	2.56	0.23	0.0072	0.22	2,191	122	3,953
30	250.13	121.18	2.41	0.23	0.0074	0.22	1,984	111	3,980
40	249.32	120.73	2.42	0.23	0.0078	0.22	1,921	107	3,973
50	249.33	120.74	2.42	0.23	0.0078	0.22	1,920	107	3,973
0	250.15	121.19	2.39	0.23	0.0110	0.21	1,334	76	5,559
10	250.08	121.15	2.48	0.23	0.0193	0.21	788	45	5,987
20	249.54	120.86	2.42	0.23	0.0209	0.21	710	41	5,963
30	249.74	120.96	2.34	0.23	0.0218	0.21	658	38	5,959
40	249.55	120.86	2.22	0.23	0.0229	0.21	596	35	5,960
50	250.15	121.19	2.40	0.23	0.0233	0.21	629	36	5,984
60	250.13	121.18	2.41	0.23	0.0240	0.21	616	36	5,971
70	250.15	121.19	2.45	0.23	0.0242	0.21	618	36	5,964

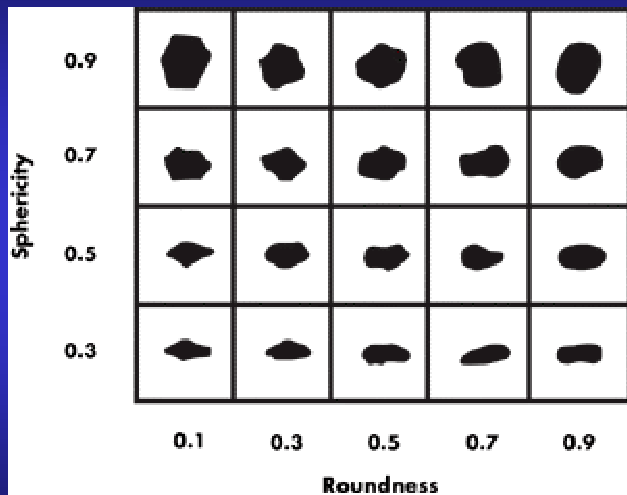
Table 5
Pre-test Sieve Analysis of Submitted Samples
From Norton Proppants, submitted 8-12-02 pm, Mike Snyder

Sample I.D.	Sample FS-081202-16 16/45		Sample FS-081202-20 16/40		Sample InterProp 20/40		Sample ValueProp 20/40	
US Standard	Weight %							
Sieve No.	Retained	Cumulative	Retained	Cumulative	Retained	Cumulative	Retained	Cumulative
8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
10	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
12	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
14	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16	5.0	5.0	0.5	0.5	0.0	0.0	0.0	0.0
18	11.1	16.1	5.6	6.1	0.0	0.0	0.0	0.0
20	14.6	30.7	24.5	30.6	4.2	4.2	4.7	4.7
25	14.7	45.4	24.1	54.7	25.4	29.6	30.1	34.8
30	18.4	63.8	23.2	77.9	50.8	80.4	46.9	81.7
35	15.8	79.6	14.4	92.3	18.8	99.2	17.0	98.7
40	12.2	91.8	7.1	99.4	0.8	100.0	1.2	99.9
45	7.0	98.8	0.5	99.9	0.0	100.0	0.1	100.0
50	1.1	99.9	0.1	100.0	0.0	100.0	0.0	100.0
60	0.0	99.9	0.0	100.0	0.0	100.0	0.0	100.0
70	0.0	99.9	0.0	100.0	0.0	100.0	0.0	100.0
80	0.0	99.9	0.0	100.0	0.0	100.0	0.0	100.0
100	0.0	99.9	0.0	100.0	0.0	100.0	0.0	100.0
pan	0.1	100.0	0.0	100.0	0.0	100.0	0.0	100.0
total	100.0		100.0		100.0		100.0	
in-size	93.8		98.9		95.8		95.2	
Median Dia. (mm)	0.683		0.721		0.664		0.671	

	Conductivity Analysis								
Start Test Date	15:01:37								
Company	Conoco Phillips								
Representative	James Carpenter								
Eng.Support No.	02-10-0753								
Cell #	WHast.	Top							
Width Core Top	9.020	Fluid	0	mls					
Width Core Bottom	9.150	Proppant	63	grams					
Width Pack, initial	0.210								
Fluid									
Additives									
		Proppant	Versa Prop				5,195	50 hour average	
Temperature	250	Type	18/40						
Closure Pressure	4000-10000	Concentration	2	lbs/ft2					
Fluid Pressure		Base line	428	Darcies	322	220	158		
Test Data	Temp	Temp	Rate	Viscosity	DP	Width	Conductivity		Closure
Time	°F	°C	mls/min	cp	psi	inches	md-ft	darcies	psi
0	268.12	131.18	3.83	0.21	0.0033	0.21	6,586	382	4,016
10	281.21	138.45	4.10	0.20	0.0051	0.205	4,241	248	3,981
0	278.60	137.00	4.10	0.20	0.0046	0.188	4,759	304	6,028
10	275.72	135.40	3.68	0.20	0.0053	0.186	3,739	241	6,036
20	277.52	136.40	3.80	0.20	0.0053	0.184	3,885	253	3,016
30	277.70	136.50	3.37	0.20	0.0057	0.184	3,152	206	6,093
40	275.49	135.27	2.86	0.20	0.0057	0.184	2,741	179	6,069
50	275.07	135.04	3.47	0.20	0.0059	0.184	3,182	208	6,078
0	275.18	135.10	3.45	0.20	0.0077	0.174	2,417	167	8,055
10	275.81	135.45	3.43	0.20	0.0090	0.174	2,058	142	8,052
20	277.16	136.20	3.44	0.20	0.0094	0.174	1,973	136	8,054
30	275.54	135.30	3.44	0.20	0.0095	0.174	1,964	135	8,040
40	275.00	135.00	3.41	0.20	0.0096	0.174	1,933	133	8,020
50	277.95	136.64	3.44	0.20	0.0100	0.174	1,852	128	8,054
0	275.40	135.22	3.41	0.20	0.0123	0.164	1,498	110	10,007
10	277.12	136.18	2.99	0.20	0.0144	0.164	1,115	82	9,971
20	276.35	135.75	3.07	0.20	0.0151	0.164	1,096	80	10,005
30	278.20	136.78	2.80	0.20	0.0151	0.164	993	73	9,956
40	277.54	136.41	2.78	0.20	0.0157	0.164	952	70	9,968
50	276.89	136.05	2.80	0.20	0.0158	0.164	951	70	9,971

Sphericity and Roundness

- Sphericity-the measure of how close a particle of proppant approaches a sphere.
- Roundness-the measure of the relative sharpness of the grain corners
- Krumbien and Sloss (1963)Chart
  - 20 individual grains judged and averaged
  - Sphericity should be greater than 0.6
  - Roundness should be greater than 0.6


Sphericity
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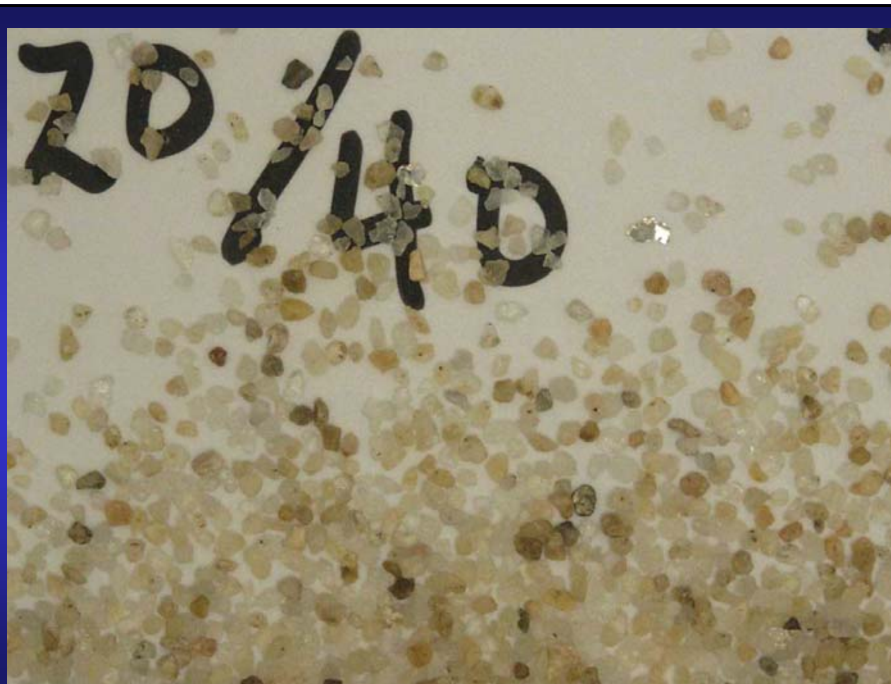
0.7
0.5
0.3
0.1
0.3
0.5
0.7
0.9
Roundness


20/40

Acid Solubility

- The solubility of sand in 12% HCl 3% HF acid is an indication of the amount of undesirable contaminants; carbonates, feldspars, iron oxides, clays.
- 5 gram in 100 milliliter of 12:3% HCl:HF for a minimum of 30 minutes
- 30/50 and Larger should be less than 2% soluble
- 40/70 and Smaller should be less than 3% soluble

Turbidity

- A result of suspended clays , silt or finely divided inorganic matter.
- Light scattering techniques
- Developed from the Jackson Candle tubidimeter.
- Calibrated from Formazin polymer - Formazin turbidity units or ftu’s

Crush Resistance

- Measure of the strength of sand
- Gives the stress the proppant can be applied to.
  - Sieve out all material not on designated sieves
  - 4 lbs./ft^2 in standard crush cell
  - One minute to the target stress, hold for 2 minutes
  - As little as 5% fines can do 50% permeability damage. (Gidley SPE 24008, Lacy SPE 36421, SPE 38590)

Suggested Maximum Fines

Mesh	Load, lbs.	Stress,psi	Maximum,%
6/12	6,283	2,000	20
8/16	6,283	2,000	18
12/20	9,425	3,000	16
16/30	9,425	3,000	14
20/40	12,566	4,000	14
30/50	12,566	4,000	10
40/70	15,708	5,000	8
70/140	15,708	5,000	6

Mineralogical Analysis

- 2 samples needed
- 1- minerals
- 1-clays
- Report all constituents over 1%

API Recommended Practice 60

- The purpose of these recommended practices is to provide standard procedures for evaluating high strength proppants.
  - Econoprop
  - Carbolite
  - Carboprop
  - VersaProp
  - Interprop
  - Bauxite
  - Sinterlite
  - Sinterball

Sand Sampling

Sampling Device - slot 8 inches x 6 inches x 4 inches

Sample Splitter - Sample reduction
Representative sample

API Requirements
3 samples per truck
a minimum of 1 sample per 20,000 lbs.
samples are combined, split and a single sample is tested

Sieve Analysis

- 6 sieves plus a pan
- Split sample of approximately 100 grams
- Add sample to the top sieve place on a Rotap for 10 minutes
- Calculate wt.% held on each sieve
- The cumulative weight should be within 0.5% of the initial weight.

ISO 2003

Minimum 7 Sieves plus the pan
Calculate median particle diameter

Sieve Sizes

Sieve opening sizes µm	3350/1700	2360/1180	1700/1000	1700/850	1180/850	1180/600	850/425	600/300	425/250	425/212	212/106
US Mesh Size	6/12	8/16	12/18	12/20	16/20	16/30	20/40	30/50	40/60	40/70	70/140
Sieves	4	6	8	8	12	12	16	20	30	30	50
	6	**8**	**12**	**12**	**16**	**16**	**20**	**30**	**40**	**40**	**70**
	8	10	14	14	18	18	25	35	45	45	80
	10	12	16	16	**20**	20	30	40	50	50	100
	12	14	**18**	18	25	25	35	45	**60**	60	120
	14	**16**	20	**20**	30	**30**	**40**	**50**	70	**70**	**140**
	16	20	30	30	40	40	50	70	100	100	200
	pan	pan	pan	pan	pan	pan	pan	pan	pan	pan	pan

A minimum of 90,0 % of the tested proppant sample should fall between the designating sieve sizes, (*i.e.*, 12/20, 16/20, 16/30, 20/40, etc*)*. A minimum of 90% of the tested proppant sample should pass the coarse designated sieve and be retained on the fine designated sieve. (i.e, 12/20, 20/40, 40/60, etc*)*. Not over 0,1 % of the total tested proppant sample should be larger than the first sieve size in the nest specified in Table 4.1 and not over 2 % of the total tested proppant sample should be smaller than the last designating sieve sizeian diameter of each grade should be made available.

Proppant Size:	1=20/40	2=16/30Sand or16-18/20-30Ceran	3=12/20 or 30/50 or12/18	4=8/12 or highcut 2 sand or 16-18/20-30
Variation	20/40 C-Lite	20/40 C-Lite	20/40Econoprop	20/40Econoprop
Proppant ----------	2.0lb/sqft-330F	2.0lb/sqft-330F	2.0lb/sqft-330F	2.0lb/sqft-330F
ENTER -->	*30*	*30*	*31*	*31*
Proppant Size				
ENTER -->	*1*	*1*	*1*	*1*
Proppant Concentration (lb/sqft)				
ENTER -->	*2*	*2*	*2*	*2*
Enter Closure pressure in psi				
ENTER -->	*11000*	*11000*	*11000*	*11000*
Enter Temperature in degrees Fahrenheit from 150 to 300 deg F				
ENTER -->	*330*	*330*	*330*	*330*
Enter Formation Modulus in millions of psi: for example, 0.5, 1 or 5 (common)				
ENTER -->	*2*	*2*	*2*	*2*
Enter Frac Fluid Damage Factor as %Retained Permeability or enter 100 and calculate on lines				
ENTER -->	*100*	*100*	*100*	*100*

Sieve	---	Percent on each sieve	--------------------	
6	0.0	0.0	0.0	0.0
8	0.0	0.0	0.0	0.0
10	0.0	0.0	0.0	0.0
12	0.0	0.0	0.0	0.0
14	0.0	0.0	0.0	0.0
16	0.1	0.1	0.0	0.0
18	0.0	0.0	0.0	0.0
20	7.9	7.9	3.7	3.7
25	48.2	48.2	28.6	28.6
30	42.4	42.4	31.4	31.4
35	1.2	1.2	34.3	34.3
40	0.2	0.2	2.0	2.0
45	0.0	0.0	0.0	0.0
50	0.0	0.0	0.0	0.0
60	0.0	0.0	0.0	0.0
70	0.0	0.0	0.0	0.0
80	0.0	0.0	0.0	0.0
100	0.0	0.0	0.0	0.0
pan	0.0	0.0	0.0	0.0
Total %	100	100	100	100

Median Phi	0.468	0.468	0.631	0.631
Median D(mm)	0.723	0.723	0.646	0.646
Median D(in)	0.028	0.028	0.025	0.025
Phi84-phi50 (Std I	0.166	0.166	0.231	0.231
Porosity (%) at 20	42.7	42.7	42.6	42.6
APIRP 56 Physical Properties				
Sphericity	0.8	0.8	0.8	0.8
Roundness	0.8	0.8	0.8	0.8
Acid Solubility %	1.7	1.7	1.9	1.9
Turbidity	20.0	20.0	161.0	161.0
Sp. Gravity	2.73	2.73	2.65	2.65
Bulk Density g/cc	1.6	1.6	1.6	1.6
lb/cuft	102.0	102.0	96.0	96.0
Crush 2000	#N/A	#N/A	#N/A	#N/A
3000	#N/A	#N/A	#N/A	#N/A
4000	20.9	#N/A	#N/A	#N/A
5000	#N/A	#N/A	1.0	1.0
Ceramics 7500	4.3	4.3	4.7	4.7
10000	12.1	12.1	13.3	13.3
12500	21.4	21.4	23.3	23.3
15000	29.8	29.8	32.1	32.1
Sieve Factor	1.00	1.00	1.00	1.00
k @P&T	83	83	70	70
Width @ 2 lb	0.227	0.227	0.230	0.230
Ideal Width	0.235	0.235	0.250	0.250
Width Corrections (a*stress)+b:				
a	1.46E-06	1.46E-06	2.22E-06	2.22E-06
b	6.40E-04	6.40E-04	3.50E-04	3.50E-04
Delta Width	0.033	0.033	0.050	0.050
Actual width	0.2019	0.2019	0.2005	0.2005
Cond @P&T	1404	1404	1169	1169
EMBEDMENT CALCULATIONS				
emb factor	0.0206	0.0206	0.0206	0.0206
emb w (in)	0.0149	0.0149	0.0133	0.0149
spalling(in)	0.0105	0.0105	0.0105	0.0105
Tot loss	0.0253	0.0253	0.0237	0.0253
eff width	0.1766	0.1766	0.1767	0.1751
Loss Ratio	0.8924	0.8924	0.9050	0.8987
Perm w/emb	74	74	63	63
Cond w/emb	1253	1253	1058	1050
Perm and conductivity with embedment and gel damage at temp and closure				
k (Darcies)	74	74	63	63
Cond (md-ft)	1253	1253	1058	1050

RP 60 Roundness and Sphericity

ISO

A.1 Sphericity and roundness

Hydraulic fracturing sand proppant, resin-coated sand proppant and gravel packing sand proppant shall have an average sphericity of 0,6 or greater and an average roundness of 0,6 or greater.

Ceramic proppants and resin coated ceramic proppants shall have an average sphericity of 0,7 or greater and an average roundness of 0,7 or greater.

API RP 60 Acid Solubility

- 7.1 General
  - a test to determine the acid solubility in acid of high-strength proppant has not been included in this standard because of the insufficient data upon which to base a recommendation…

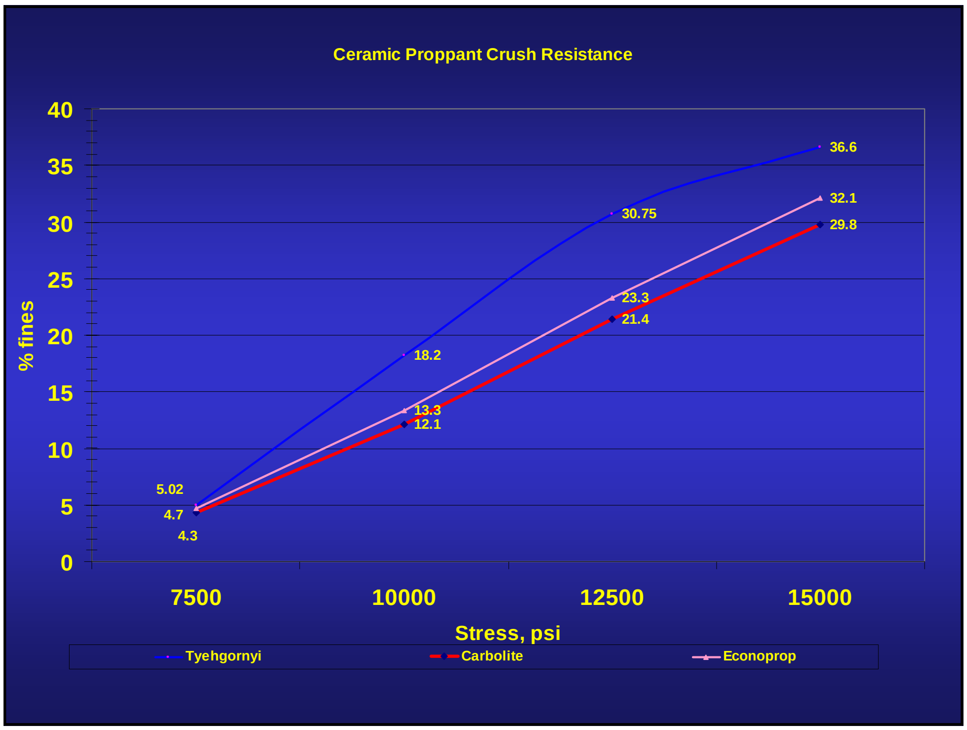
ISO 2003

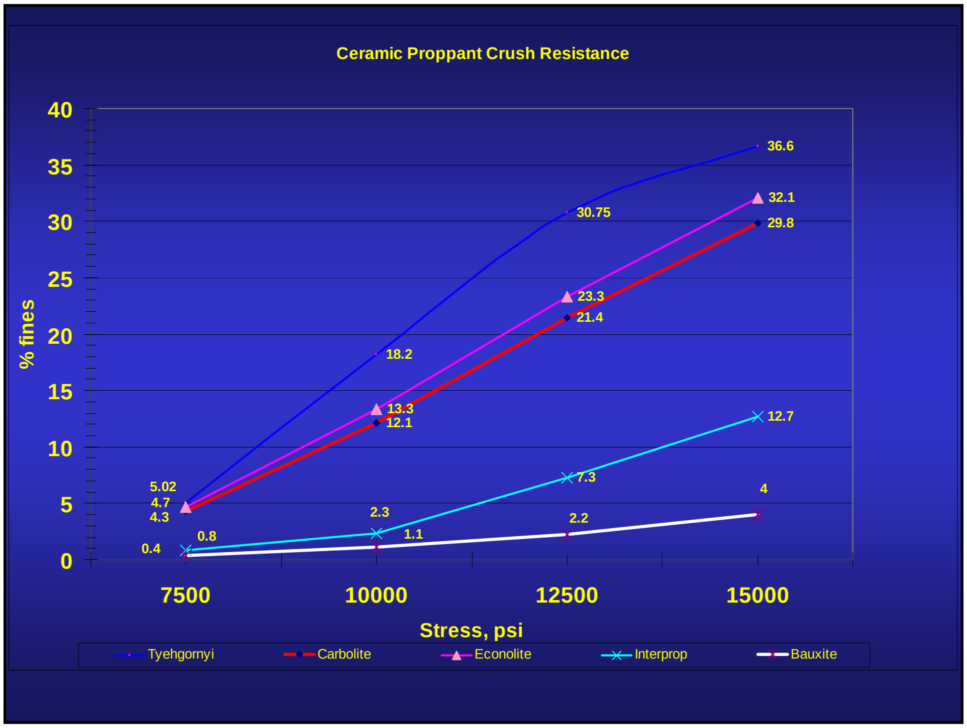
- Table — Maximum acid solubility

- weight % in 12%HCl:3%HF Acid
- Hydraulic fracturing sand, resin coated sand, gravel packing sand proppants
- Larger than or equal to 30/50 2,0
- Smaller than 30/50 3,0
-
-
- Ceramic proppants and resin coated ceramic proppants 7,0

API RP 60 Crush Resistance

- Used to determine the stress at which a proppant produces excessive fines.
- 7,500 psi
- 10,000 psi
- 12,500 psi
- 15,000 psi
- designed to indicate the maximum stress the material should be subjected to.





Suggested Maximum Fines For API RP 60

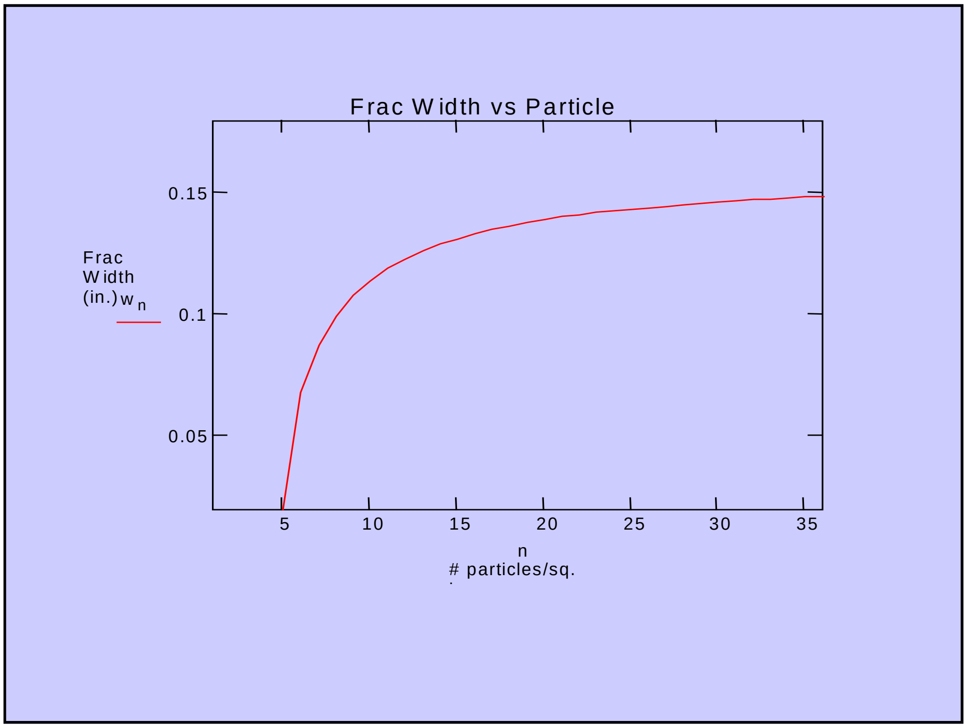
Size	Suggested Maximum
• 12/20	25
• 16/20	25
• 20/40	10
• 40/70	8

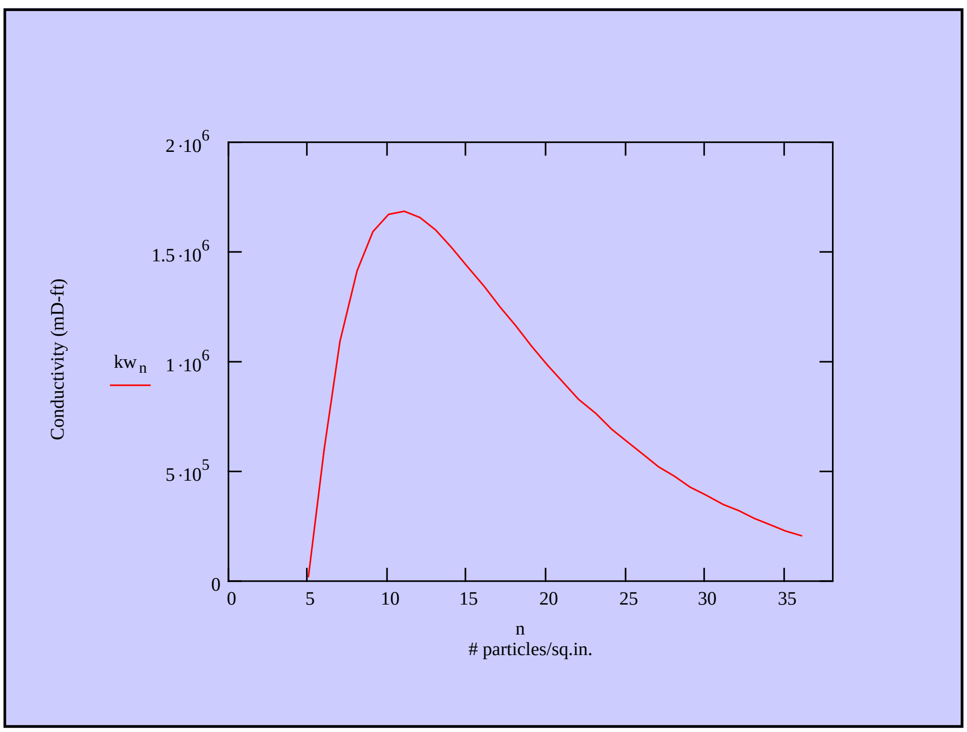
ISO Suggested Maximum Fines

Mesh size	Load on cell kN (lb_f)	Stress on proppant MPa (psi)	Suggested maximum crushed material%
6/12	28,0 (6 283)	13,8 (2 000)	(18,0) 20,0
8/16	28,0 (6 283)	13,8 (2 000)	(14,0) 18,0
12/20	42,0 (9 425)	20,7 (3 000)	16,0
16/30	42,0 (9 425)	20,7 (3 000)	14,0
20/40	56,0 (12 566)	27,6 (4 000)	(10,0) 14,0
30/50	56,0 (12 566)	27,6 (4 000)	10,0
40/70	70,0 (15 708)	34,5 (5 000)	8,0
70/140	70,0 (15 708)	34,5 (5 000)	6,0
New size data			

Bulk Density, Apparent Density and Absolute Density

- Bulk Density-describes the weight of the proppant that will fill a unit volume.
  - Sand kings
  - fractures
- Absolute density-excludes internal/interconnected porosity
- Apparent density-includes internal/interconnected porosity


Frac Width vs Particle
0.15
0.1
0.05
Frac
Width
(in.) w n
5
10
15
20
25
30
35
n
particles/sq.


2·10^6
1.5·10^6
1·10^6
5·10^5
0
Conductivity (mD-ft)
kw n
0
5
10
15
20
25
30
35
n
particles/sq.in.


The End

Geological Services & Special Core Analysis: Capabilities & Solids Analysis

Section 5
2006

Printed: 4/26/2007

Overview Of Geological Services & Special Core Analysis Groups

- Functions
- People
- Analytical capabilities
- Instrument capabilities
- Materials characterization
- Formation evaluation projects
- Lab Tours (GS, Geomech, SCAL)
- Solids analysis - discussion

Geological Services & Special Core Analysis Functions

- Support operations, sales, and engineering personnel by providing geological data, interpretations, and completion engineering expertise
- Support product and engineering research by providing application tests such as core flow studies, and material identification & characterization

Geological Services People

- Dr. Gerald (Gerry) Braun
  - Geologist, Mineralogist
  - 32 Years Oilfield Experience
- BJ Davis
  - Geologist, Flow Studies Analyst
  - 24 Years Oilfield Experience
- Laura Vestal
  - Geologist, 1.5 years Oilfield Experience
- Amanda Seholm
  - Geological Technician

Special Core Analysis People

- Carolyn DeVine
  - Geologist, Geochemist, Flow Studies Analyst
  - 23 Years Oilfield Experience
- Jennifer Cutler
  - Flow Studies Analyst
  - 24 Years Oilfield Experience
- Kimberley Spurlock
  - Flow Studies Analyst
- Vicki Johnson
  - SCAL Technician

Slide 5

EDC, Tomball, TX

Analytical Capabilities

- Research & Geological consultation
- Rock-pore network characterization
  - SEM, XRD, XRF, thin section
  - Interpretation of potential completion problems
  - Recommendations for completion & stimulation
- Core flow studies
  - Short- & long-core computerized systems
  - Rock-fluid compatibility
- Materials characterization

Slide 6

EDC, Tomball, TX

Instrument Capabilities

- Stereomicroscopy
- SEM / EDS and ESEM
- Thin section petrography
- X-ray diffraction
- X-ray fluorescence
- Porosity & permeability measurement
  - Gas & liquid
- Special core analysis
  - Gas & liquid regained perms

Materials Characterization (Solids Analysis)

- Precipitated tubing scales
- Produced solids
- Bailed solids
- Filtered solids
- Emulsion solids

Materials Characterization
(Solids Analysis)

Slide 9

EDC, Tomball, TX

Formation Evaluation Projects
"Understand The Reservoir First"

- Reconnaissance reservoir study used to understand:
  - Formation evaluation requirements.
  - Critical drilling / completion issues.
  - Stimulation / production outcomes.

Slide 10

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"Understanding The Reservoir First"

- Geological and engineering data.
  - Review published literature
    - SPE, AAPG, internet
  - Operator in-house geological reports
  - Formation information (BJS Archives)
- Determine if any new laboratory analysis is needed
- Discuss objective(s) with internal and external clients

Slide 11

EDC, Tomball, TX

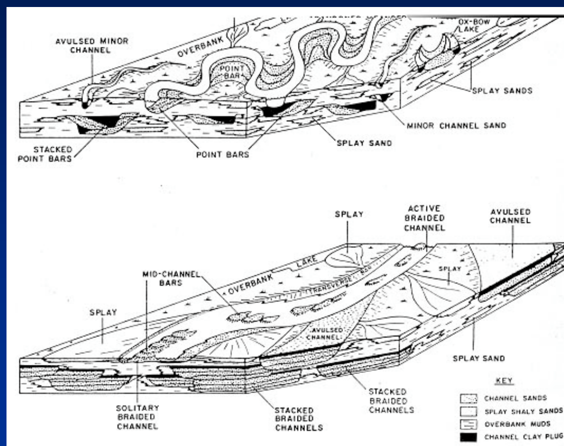
Formation Samples

- Full diameter cores
- 1" & 1½" diameter core plugs
- Rotary sidewall core plugs
- Well drill cuttings
- Percussion sidewalls
- "Produced" formation samples

Slide 12

EDC, Tomball, TX

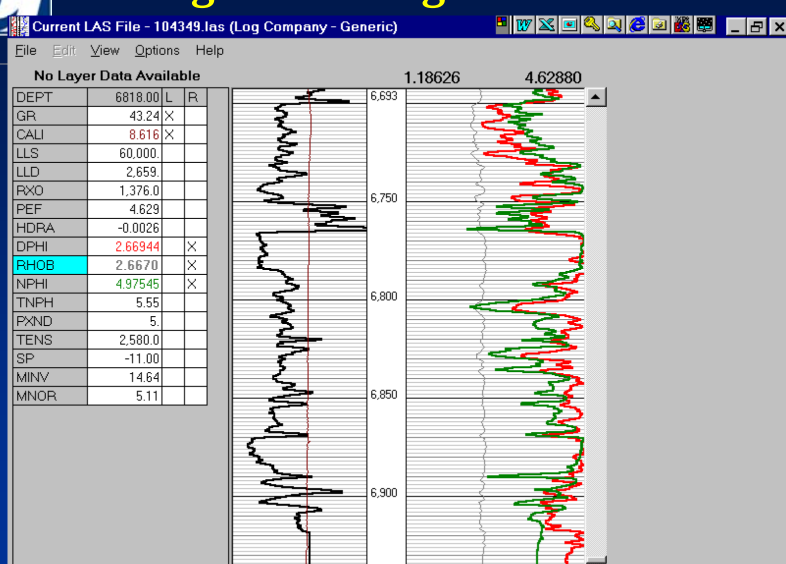
Fluvial-Deltaic Depositional Systems

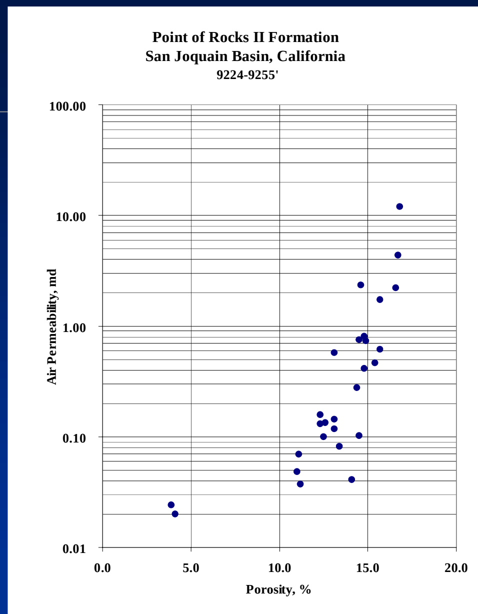


Schematic block model of A) meandering and B) braided channel systems illustrating lateral and vertical relationships among building blocks. (No scale implied).

Source: Models of Meandering and Braided Fluvial Reservoirs with Examples From the Travis Peak Formation, East Texas., Davies, et al., SPE 24692, 1992.

Engineer's Log Assistant





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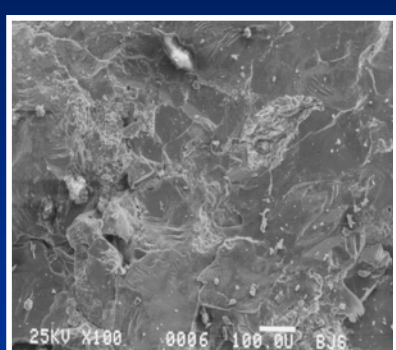
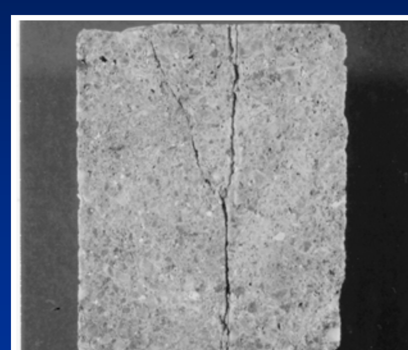
Natural Fractures in Morrow Core, Eddy County





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Natural Fracture Surface Quartz and Calcite Crystals

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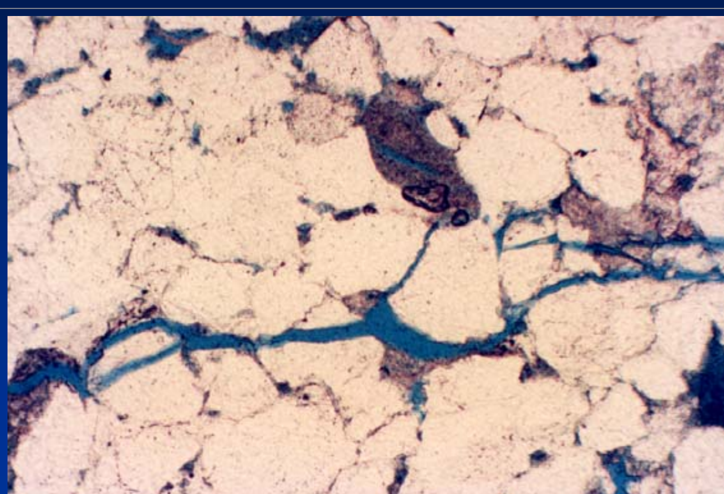
EDC, Tomball, TX

Fracture In Thin Section



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Mineralogical Data

Table #2 - Mineralogical Analysis (XRD & XRF Results)

	Minerals	13345.5'	13354.5'	13357.6'	13359.4'
Framework Grains	Quartz (SiO_2)	82%	80%	83%	80%
	Plagioclase Feldspar	6	7	5	7
Carbonates	Calcite ($CaCO_3$)	2	2	1	2
	Ankerite ($Ca[Mg_{0.67} Fe_{0.33}][Co_3]_2$)	1	1	1	1
Sulfides	Pyrite(FeS_2)	trace	trace	trace	trace
Clays	Mica + Illite	trace	1	---	1
	Mixed-Layer $Illite_{90}/Smectite_{10}$	8	7	8	7
	TOTALS	100%	100%	100%	100%
ACID SOLUBILITY TESTING					
15% HCl Solubility		4.7 %	4.2 %	4.1 %	4.2 %
Soluble Iron Content (%)		0.20%	0.17%	0.15%	0.15%

Review of the air-dried and glycol-solvated clay slides indicates that these mixed-layer illite/smectite clays are composed of **approximately 90% illite layers**.

Slide 19

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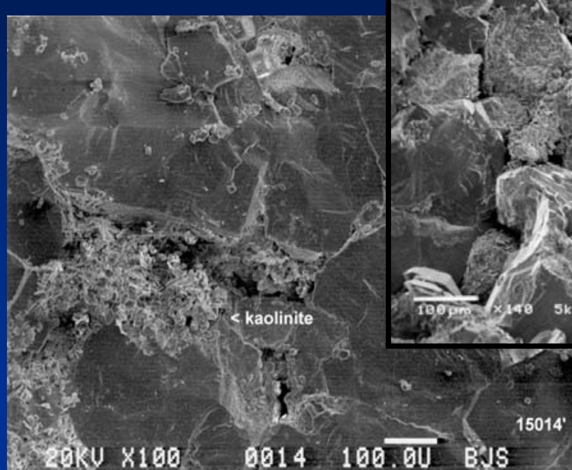
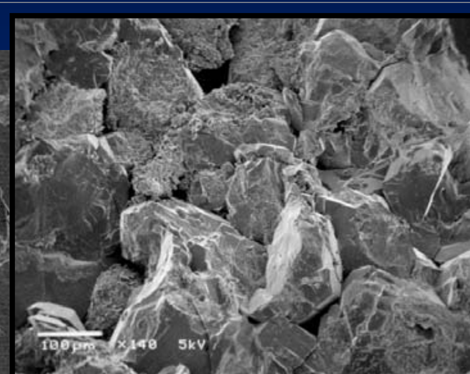
Overviews of Sandstones





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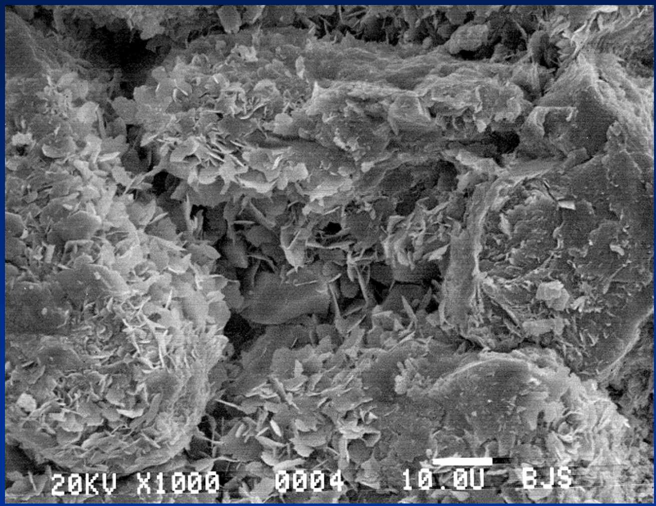
Chlorite



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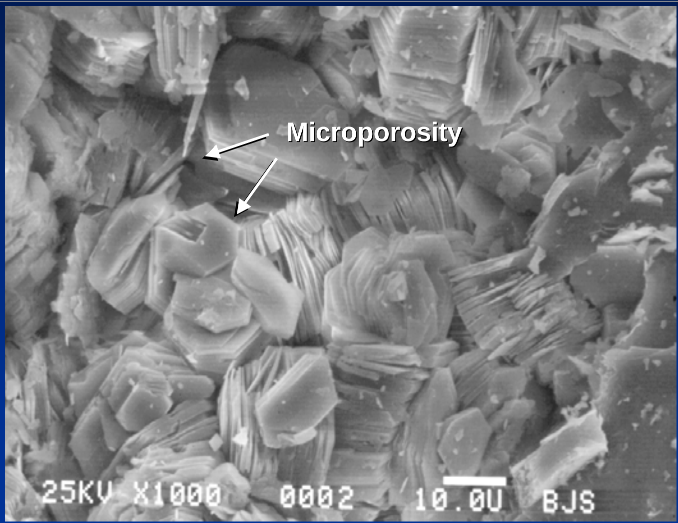
Kaolinite



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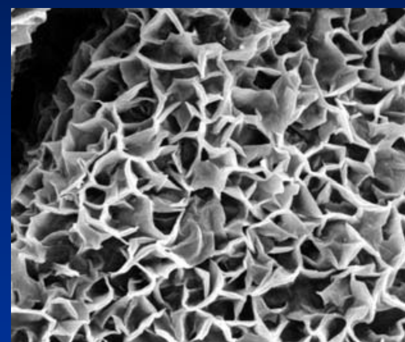
Montmorillonite Group

- Diagenetic or detrital
- Detrital
  - Shales & shale clasts
  - Laminations in Sandstones
- Diagenetic
  - Grain-coating
  - Pore-bridging



Slide 23

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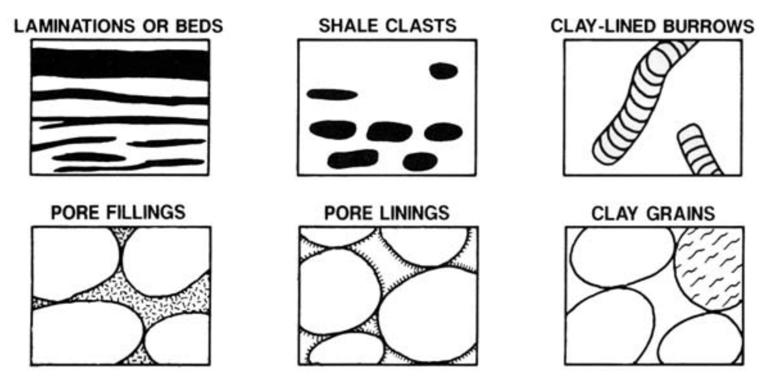
Clay Occurrences (1 of 2)



Modified from Moore et. al., 1993

DETERMINED FROM VISUAL EXAMS (STEREOMICROSCOPE, SEM, THIN SECTION)

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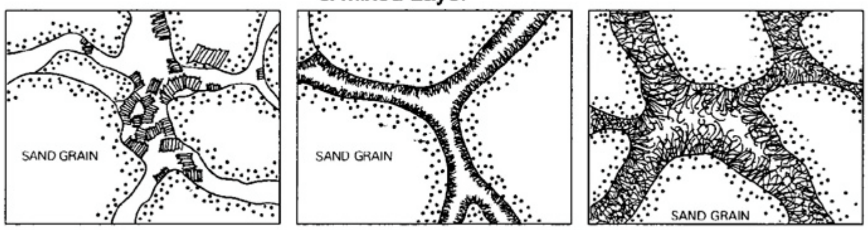
Clay Occurrences (2 of 2)

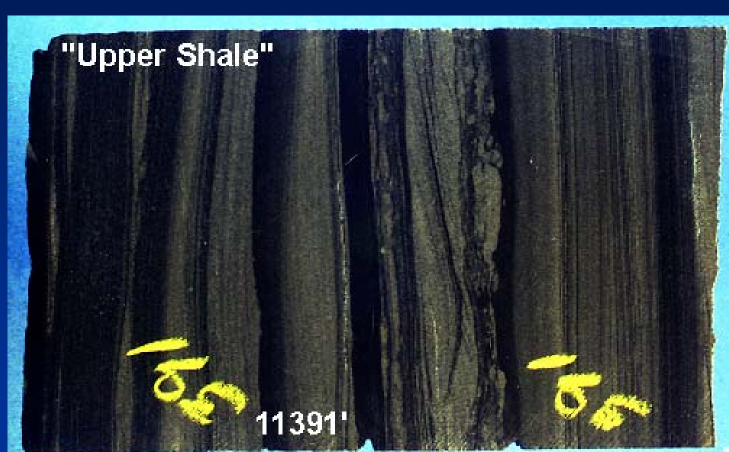
Discrete Particle - Kaolinite and Chlorite

Pore Lining - Illite, Chlorite, Smectite, & Mixed Layer

Pore Bridging - Illite and Chlorite



Clays in Laminations



Grain-Coating Clays

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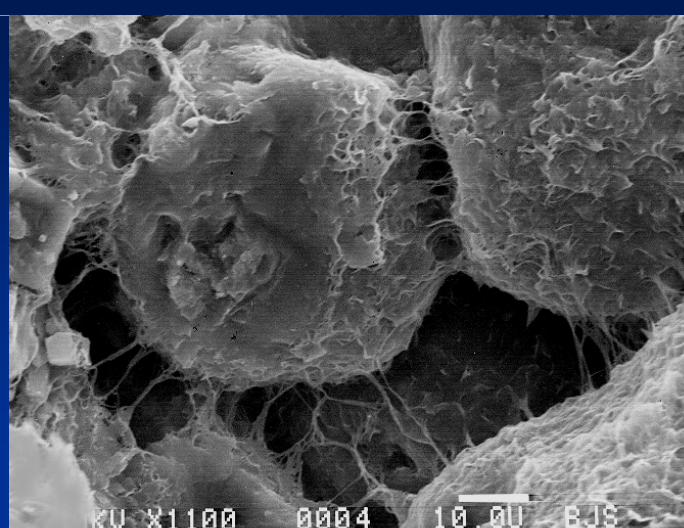
Pore-Bridging Clays



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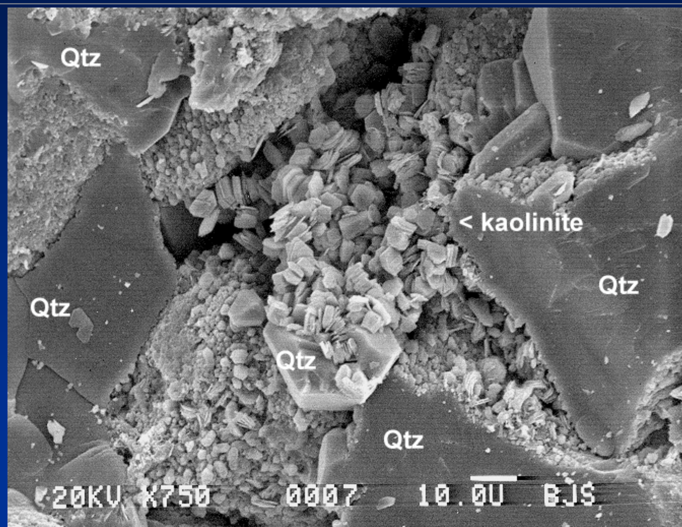
Pore-Filling Clays



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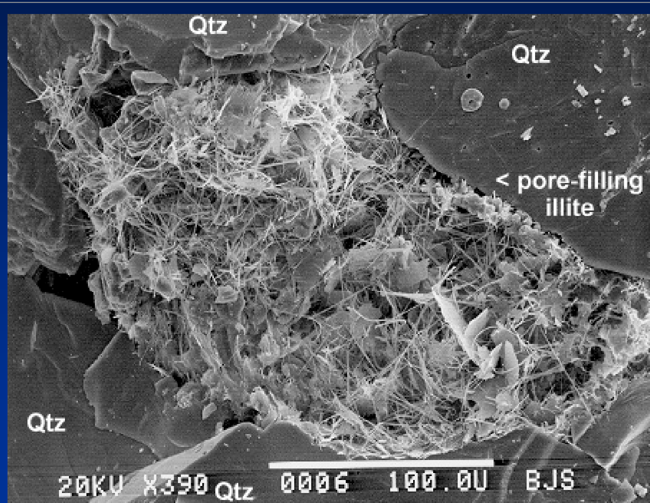
Grain-Replacing Clays



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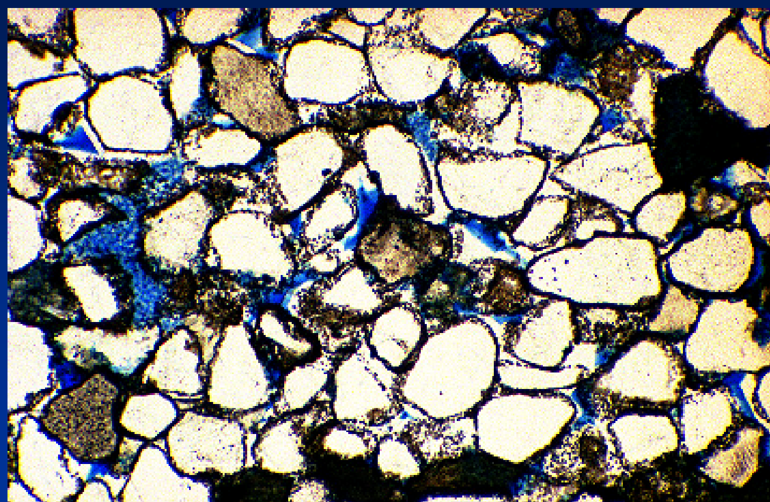
Siderite-Cemented Sandstone



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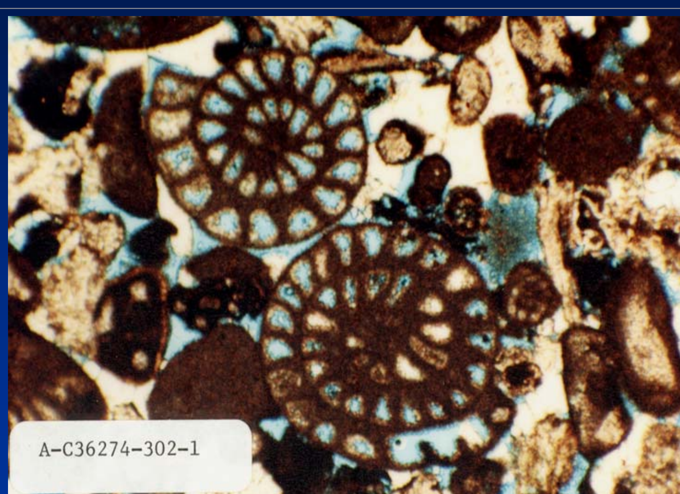
Fossiliferous Limestone



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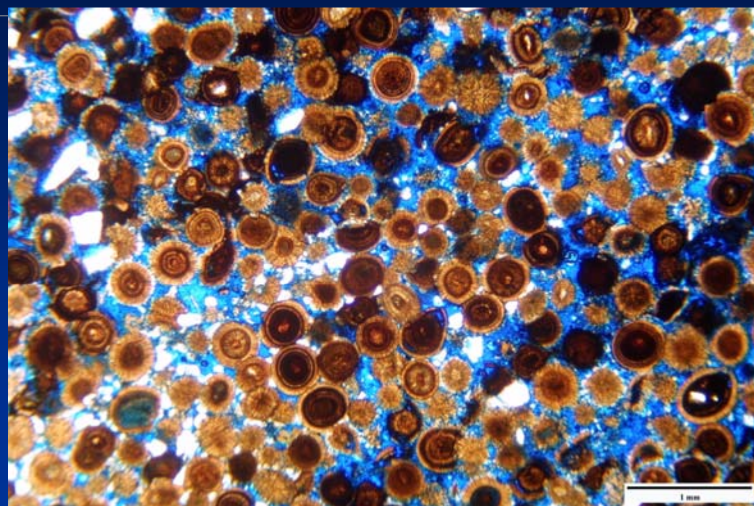
Oolitic Limestone



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Formation Evaluation Associated Tests

- Acid Solubility
- Particle Size Analysis (sieve analysis)
- Immersion Testing (rock/fluid reaction)
- Capillary Suction Time Testing
- Water Analysis
- Mechanical Rock Properties
  - Young's Modulus
  - Poisson's Ratio
  - Brinell Hardness

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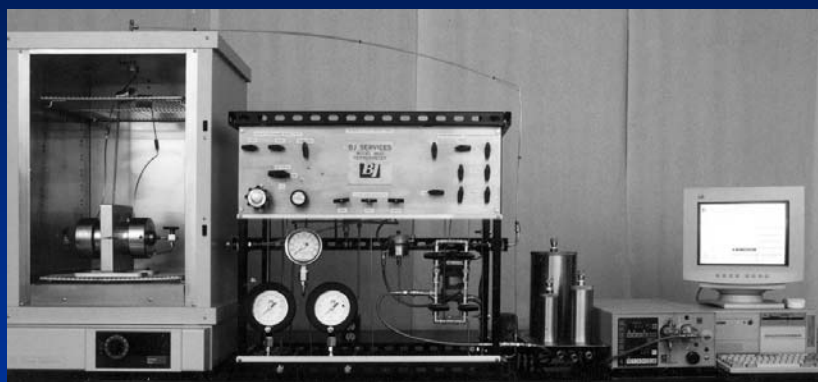
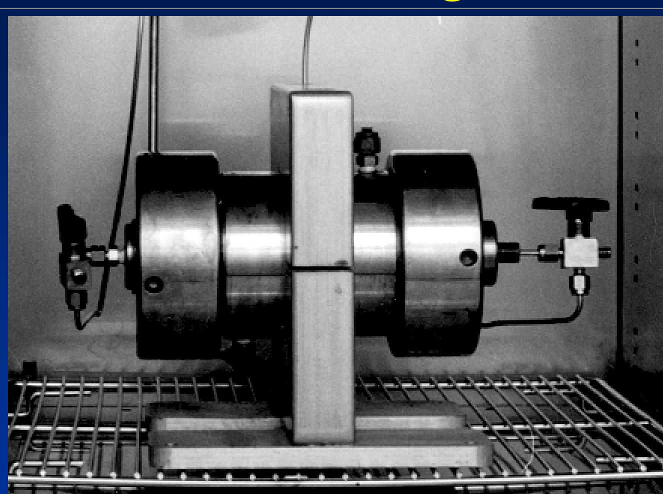
Liquid/gas Permeability Testing



Reservoir Temperature Core Testing



Potential Completion Problems

- Lack of adequate reservoir characterization
- Acid sensitive minerals
- Mud acid sensitivity
- Migratable fines (during production)
- Natural fracture leak-off (during stimulation)
- Undersaturated reservoirs causing aqueous imbibition?
- Freshwater (or low salinity brine) sensitive clays

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Stimulation Recommendations
Individual Wells

- Acid stimulation
- Hydraulic fracture treatment
  - Acid-based
  - Water-based
  - Oil-based
  - Methanol-based
  - Gas-based
- Remedial treatments

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Field & Formation Studies

Yegua	Selma Chalk	Frontier
Redfork	Morrow (Texas Panhandle)	Oriskany
Hosston	Morrow (Permian)	Spiro
Almond	North Sea Chalks	Vicksburg
Dakota	Offshore Miocene Sands	Devonian

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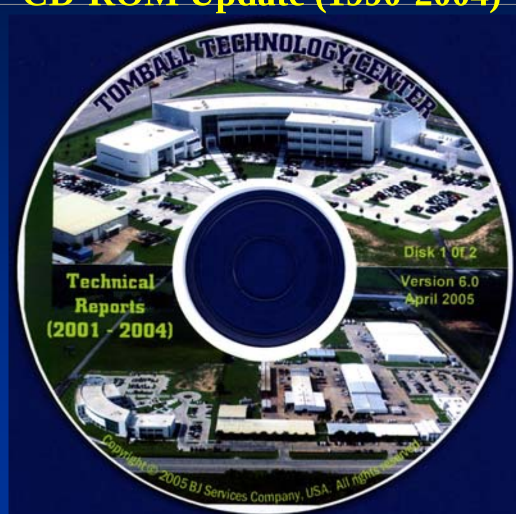
Technology Transfer
Technical Service Reports
CD-ROM Update (1990-2004)



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Lab Tours

- Geological Services Labs
  - Geological Services (2nd floor)
  - Geomechanics (1st floor)
- Special Core Analysis Lab

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Solids Analysis

- Objectives
  - #1 - Take care of the Customer
    - Internal (BJ)
    - External (operators)
  - Identify the cause of the problem.
  - Recommend an acceptable, cost-effective solution.
  - Recommend a cost-effective, preventative measure.

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Tools

- Stereomicroscope (20X preferable)
- Magnet
- Hotplate
- Torch / burner
- Centrifuge
- Separatory Funnel
- Miscellaneous glassware
- Acids (HCl, HCl/HF, etc)
- Solvents (water, xylene, acetone, hexane)

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BJ Services Chemicals

- Surfactants
- De-emulsifiers
- Mutual Solvents

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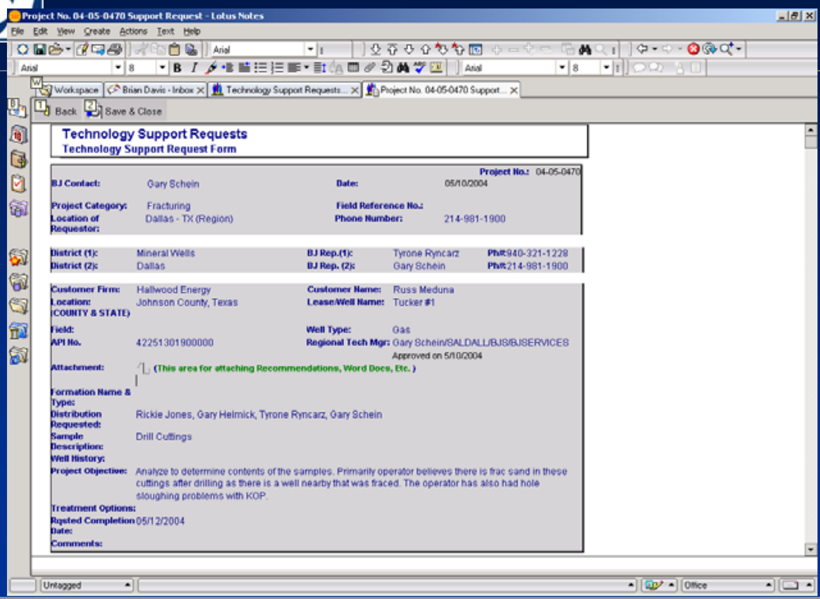
Technology Support Requests



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Ask Questions!!

- Origin of sample?
  - Obtained how?
    - Flowed back, bailed, scraped off pump, etc.
- Well History?
  - New / Old completion?
  - Prior stimulation or remedial treatment?
- Type of well?
  - Producer
  - Injector (waterflood or salt water disposal).
- Production?
  - Oil, gas, water, or combinations?

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Solids Types
Organic Solids

- Paraffins
- Asphaltenes
- Paint chips
- Rubber packers, etc.
- Fluid loss additives

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Solids Types
Combinations - Organic + Inorganic

- Emulsions (oil, water, inorganics)
- Pipe dope (grease, inorganics)

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Solids Analysis

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Solids Types
Inorganic

- Wellbore scales
  - Carbonates, iron oxides/hydroxides, sulfates, sulfides, etc.
- Proppants
- Formation materials
  - Sand, shale, fines, etc.
- Cement phases
- Drilling mud
- Phosphates
- Salts
- Perforating debris
- Amorphous solids

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Inorganic Solids

- Tubing Scale

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Inorganic Solids

- Tubing Scale

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Geological Services & Special Core Analysis Deliverables

- Single-well formation evaluation reports
- Solids analyses reports
- Field and formation studies
- Geomechanics Studies
- Core-flow studies
- Consulting and presentations
- Training

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Geological Laboratories Capabilities & Solids Analysis

Section 4

May/June 2007

Printed: 5/14/2007

Overview Of Geological Laboratories

- Functions
- People
- Analytical capabilities
- Instrument capabilities
- Materials characterization
- Formation evaluation projects
- Lab Tours (GS, Geomech, SCAL)
- Solids analysis - discussion

Geological Laboratories Functions

- Support operations, sales, and engineering personnel by providing geological data, interpretations, and completion engineering expertise

- Support product and engineering research by providing application tests such as core flow studies, and material identification & characterization

Geological Services People (1 of 3)

- Dr. Gerald (Gerry) Braun
  - Geologist, Mineralogist
  - 33 Years Oilfield Experience
- BJ Davis
  - Geologist, Flow Studies Analyst
  - 26 Years Oilfield Experience
- Laura Williams
  - Geologist, 2.5 years Oilfield Experience
- Amanda Seholm
  - Geologist, 1 year Oilfield Experience

Geological Services People (2 of 3)

Geomechanics Laboratory

- Dr. Russell Maharidge
  - Physicist - 28 years oilfield experience
- Michelle West-Egros
  - Lab Analyst

Slide 5

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Geological Services People (1 of 3)

Special Core Analysis Laboratory

- Jennifer Cutler
  - Flow Studies Analyst
  - 25 Years Oilfield Experience
- Kimberley Spurlock
  - Flow Studies Analyst
  - 6 Years Oilfield Experience
- Vicki Johnson
  - SCAL Technician

Slide 6

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Analytical Capabilities

- Research & Geological consultation
- Rock-pore network characterization
  - SEM, XRD, XRF, thin section
  - Interpretation of potential completion problems
  - Recommendations for completion & stimulation
- Core flow studies
  - Short- & long-core computerized systems
  - Rock-fluid compatibility
- Geomechanics
  - Frac Model Inputs
- Materials characterization

Slide 7

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Instrument Capabilities

- SEM / EDS and ESEM
- Thin section petrography
- X-ray diffraction
- X-ray fluorescence
- Porosity & permeability measurement
  - Gas & liquid
- Special core analysis
- Gas & liquid regained perms
- Geomechanics

Slide 8

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Materials Characterization
(Solids Analysis)

- Precipitated tubing scales
- Produced solids
- Bailed solids
- Filtered solids
- Emulsion solids

Slide 9

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Materials Characterization
(Solids Analysis)

Slide 10

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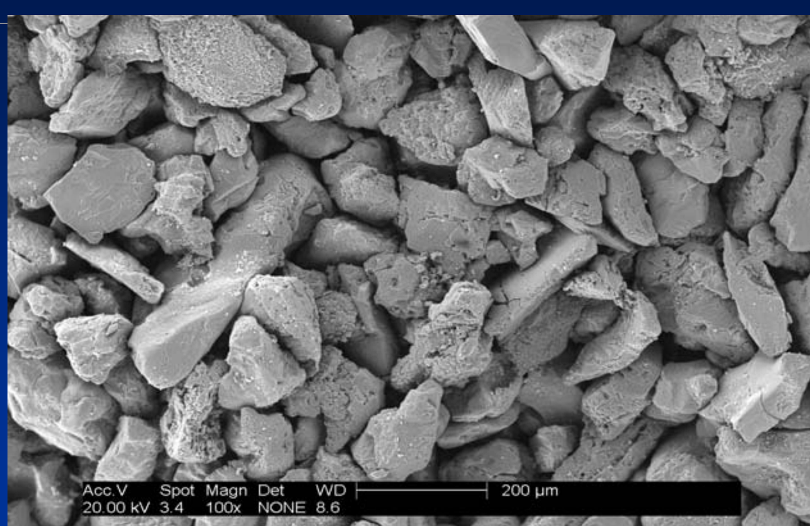
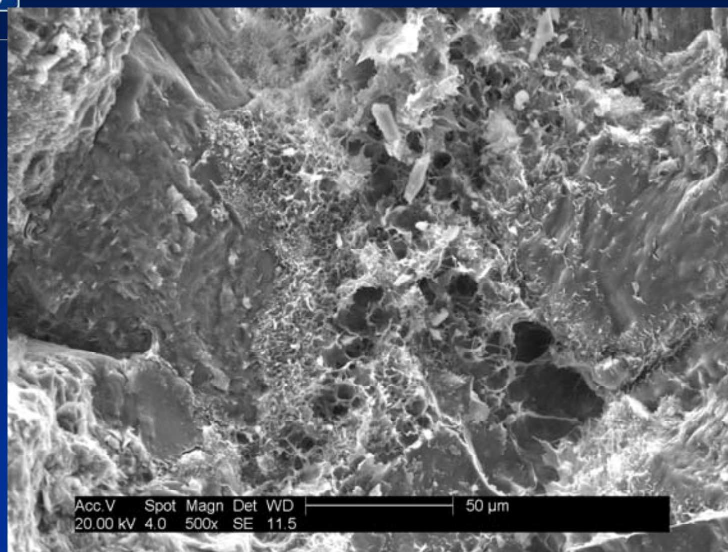
Formation Evaluation Projects
"Understand The Reservoir First"

- Reconnaissance reservoir study used to understand:
  - Formation evaluation requirements.
  - Critical drilling / completion issues.
  - Stimulation / production outcomes.

Very Good reservoir quality with minimal authigenic cements



Poor reservoir quality with abundant authigenic cements



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"Understanding The Reservoir First"

- Geological and engineering data.
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Slide 14

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Formation Samples

- Full diameter cores
- 1” & 1½” diameter core plugs
- Rotary sidewall core plugs
- Well drill cuttings
- Percussion sidewalls
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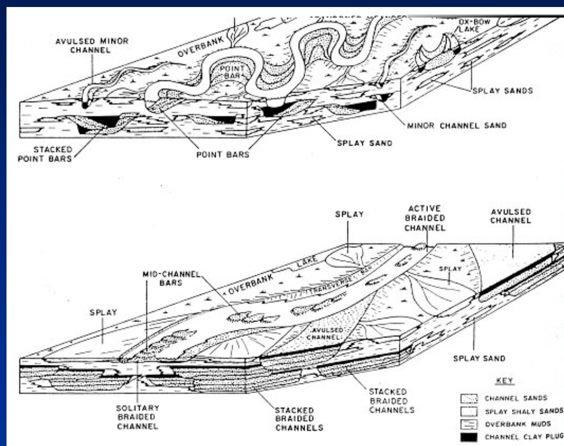
Reservoir Samples

Wholecore, coreplugs, drill cuttings

Fluvial-Deltaic Depositional Systems



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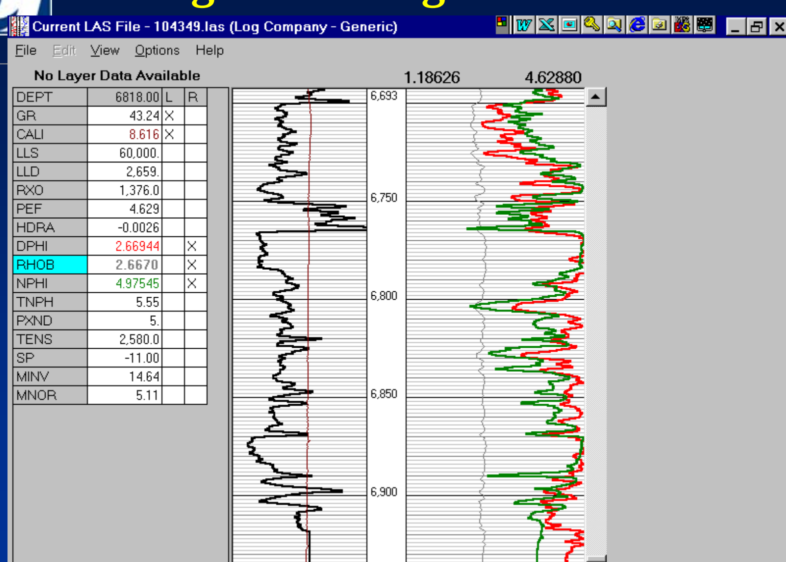
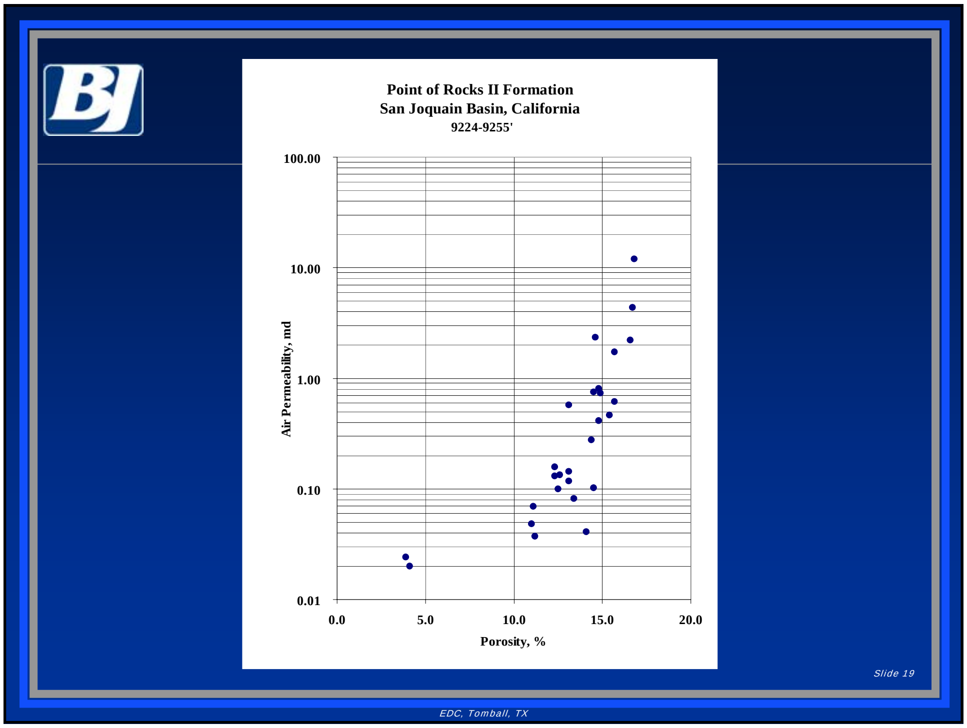
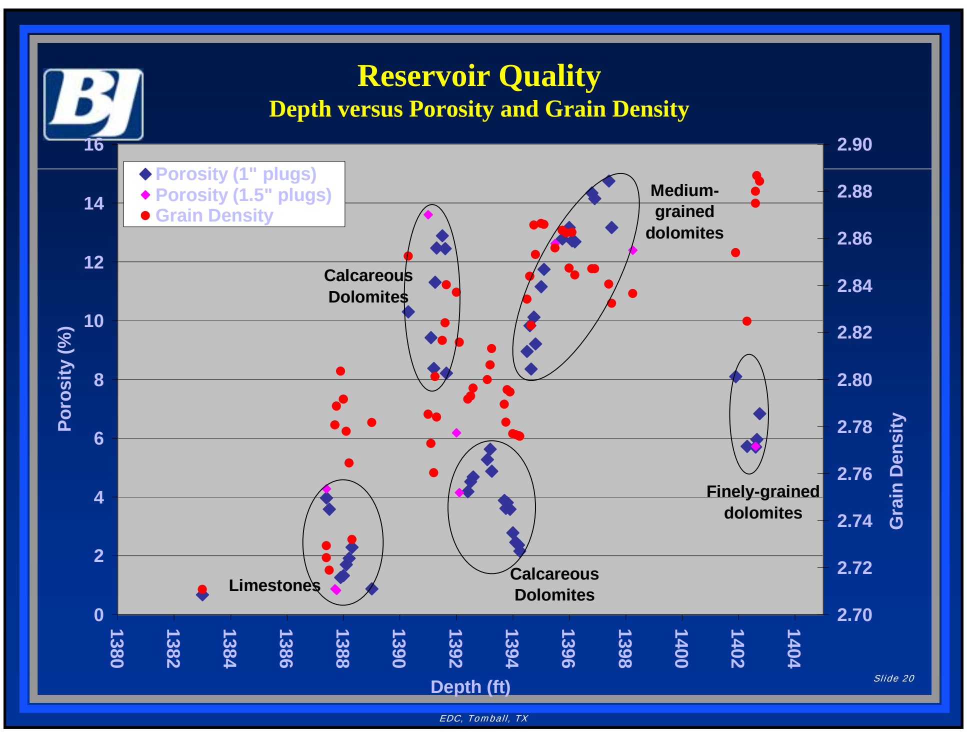
Engineer's Log Assistant





Reservoir Quality

Depth versus Porosity and Grain Density



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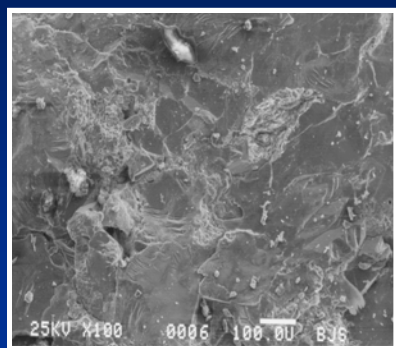

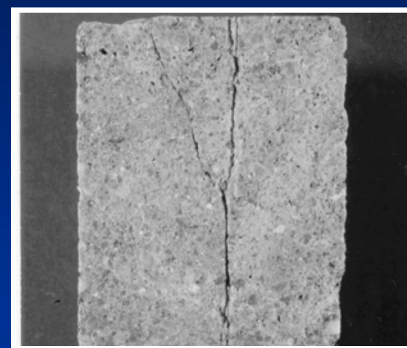

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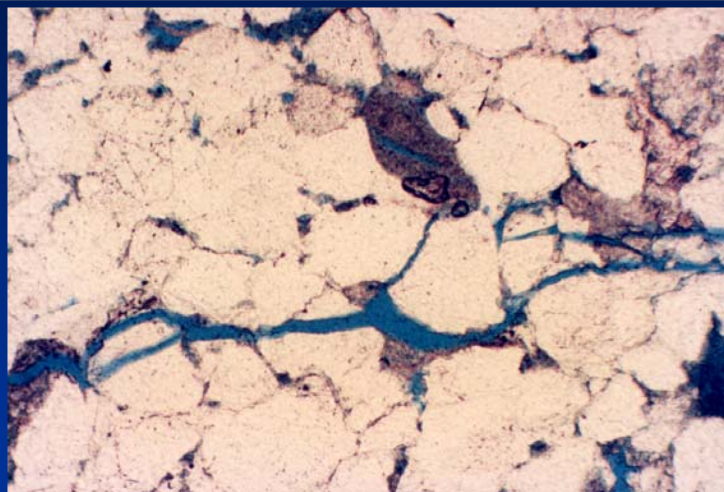
Natural Fracture Surface Quartz and Calcite Crystals

Slide 22

Fracture In Thin Section



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Slide 24

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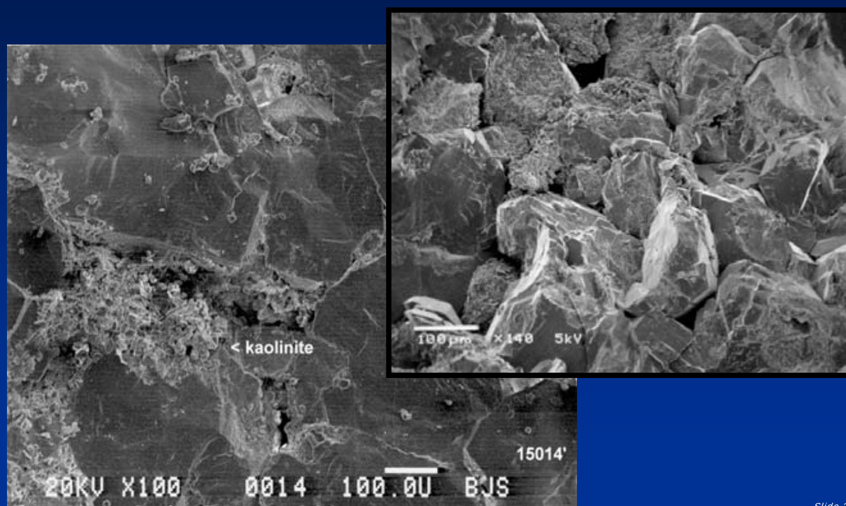
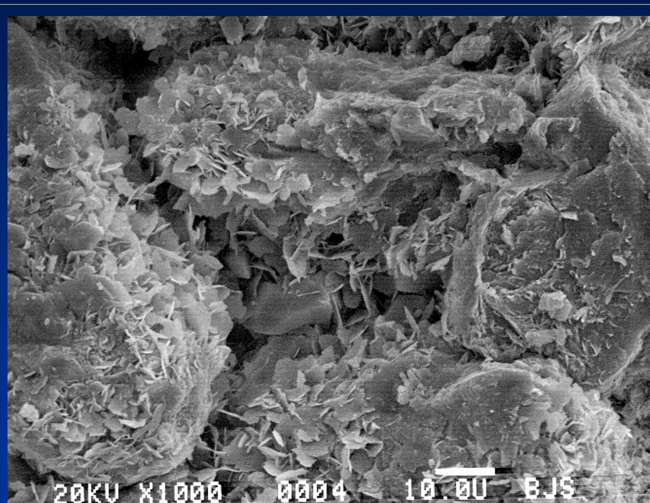
Overviews of Sandstones



Chlorite



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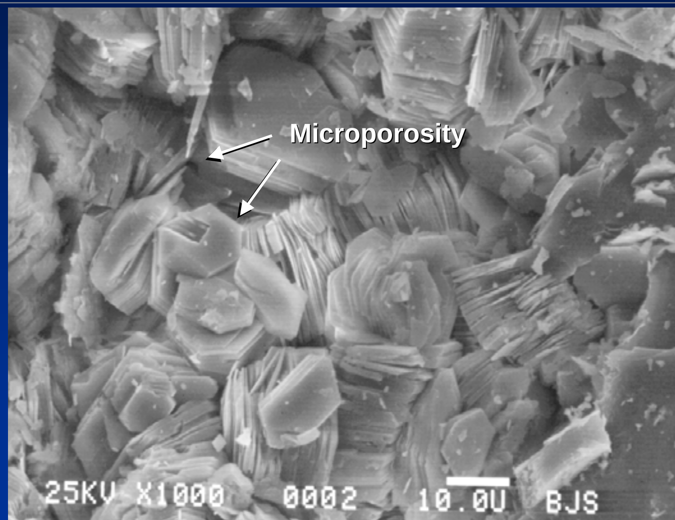

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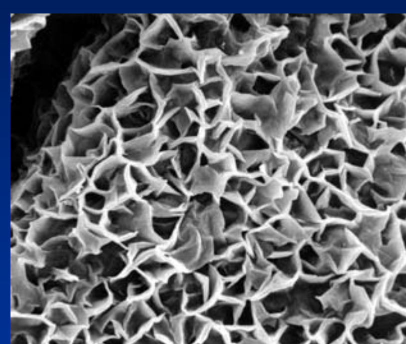
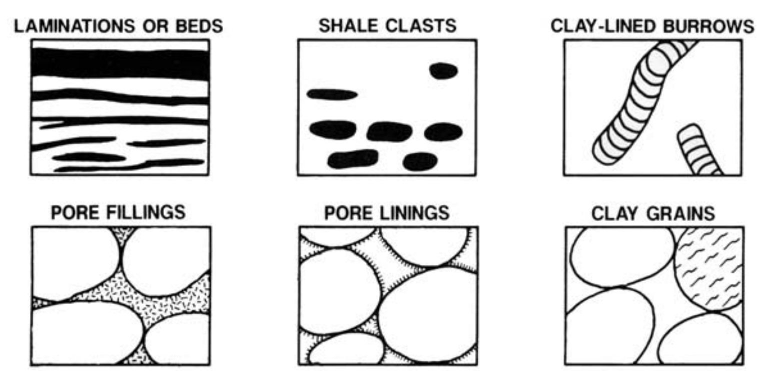


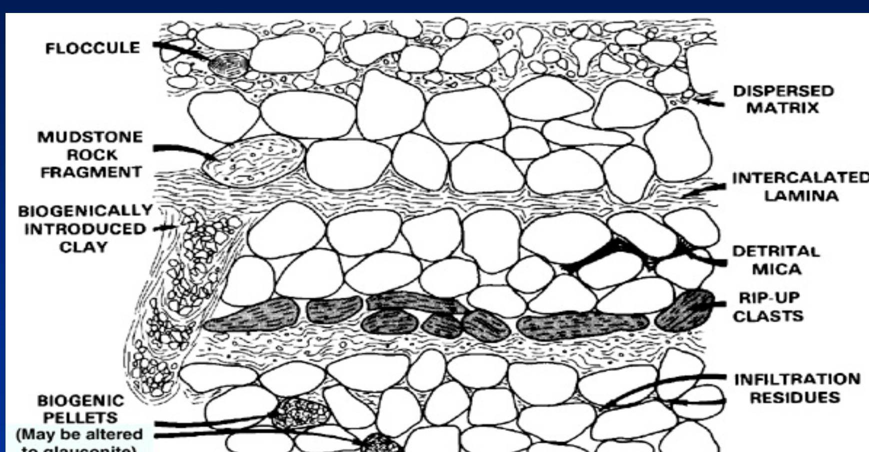
Clay Occurrences (1 of 3)



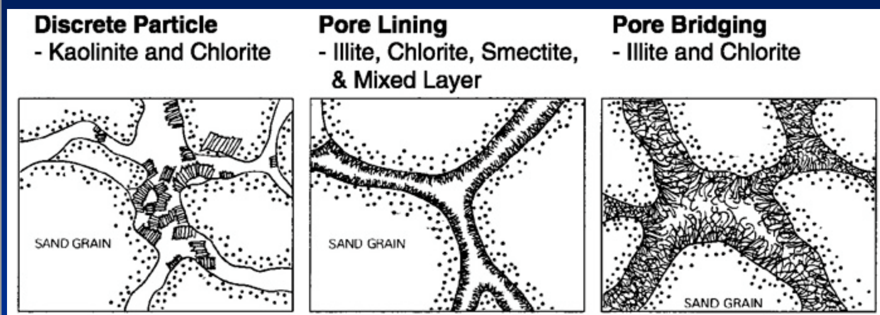
Modified from Moore et. al., 1993

DETERMINED FROM VISUAL EXAMS (STEREOMICROSCOPE, SEM, THIN SECTION)

Detrital Clay Occurrences (2 of 3)



Clay Occurrences (3 of 3)



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Clays in Laminations

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Grain-Coating Clays

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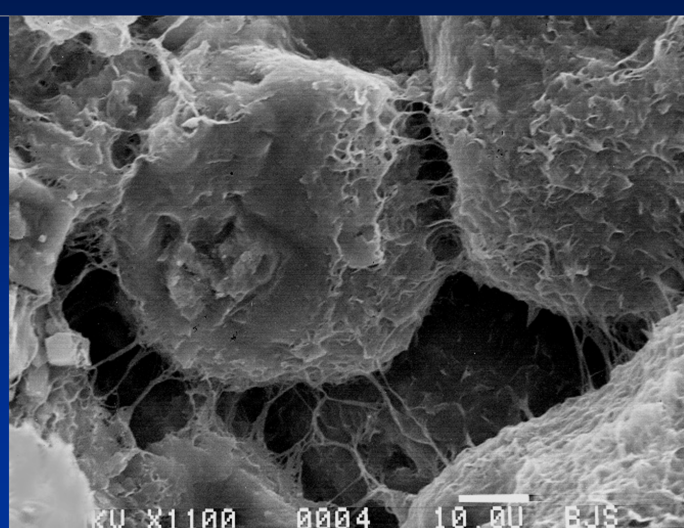
Pore-Bridging Clays



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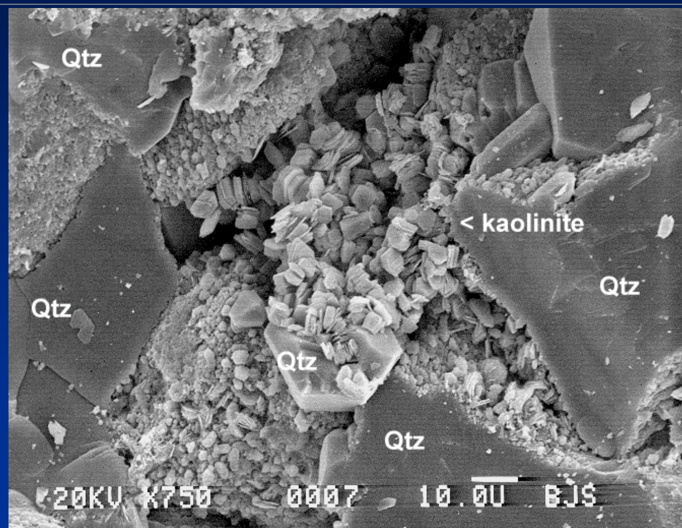
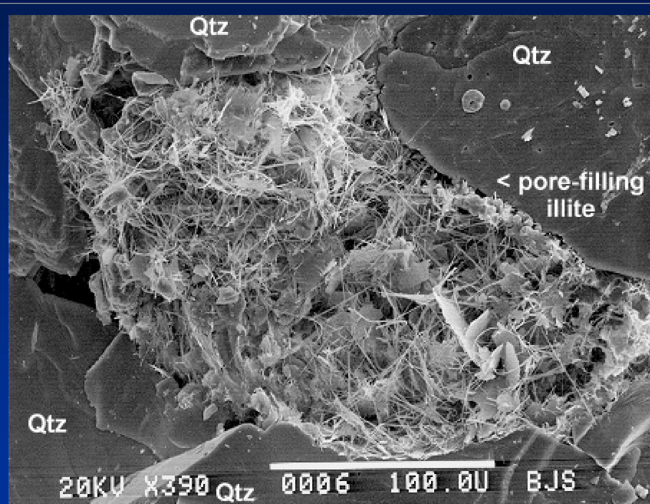
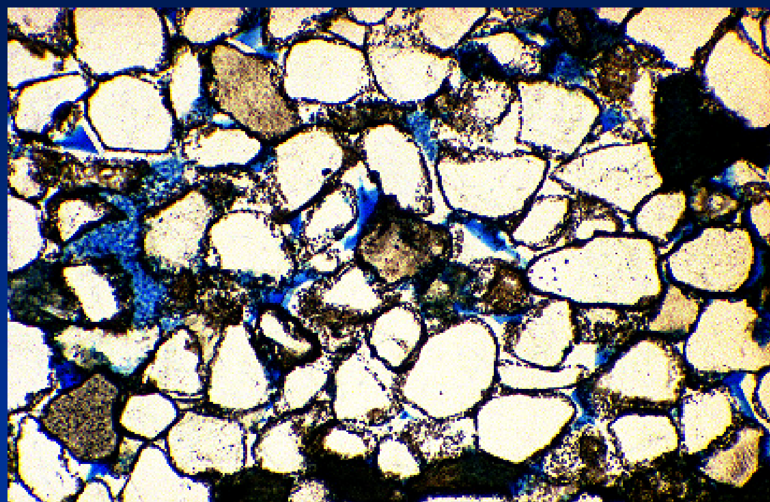
Pore-Filling Clays



Grain-Replacing Clays



Siderite-Cemented Sandstone



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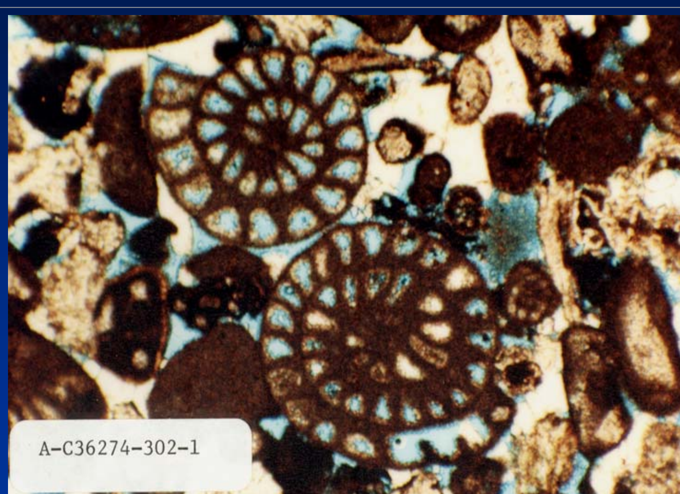
Fossiliferous Limestone



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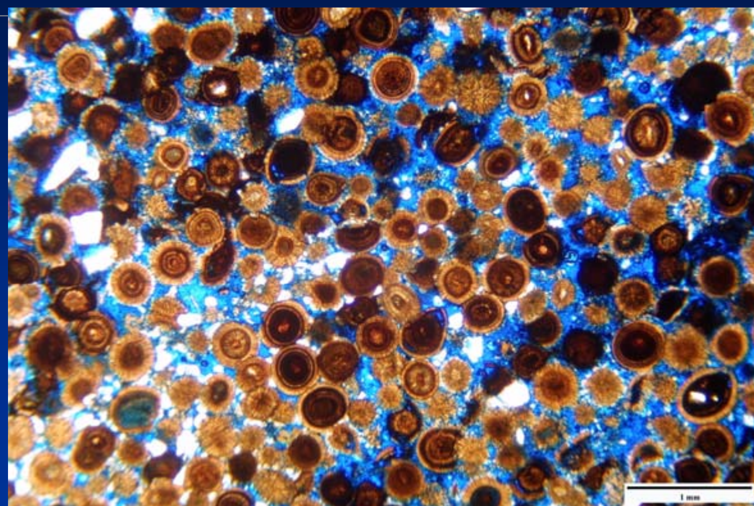
Oolitic Limestone



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Formation Evaluation Associated Tests

- Acid Solubility
- Particle Size Analysis (sieve analysis)
- Immersion Testing (rock/fluid reaction)
- Capillary Suction Time Testing
- Water Analysis
- Mechanical Rock Properties
  - Young’s Modulus
  - Poisson’s Ratio
  - Brinell Hardness

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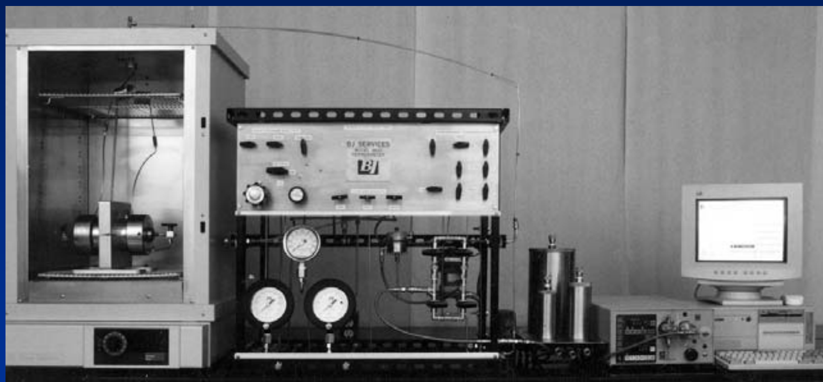
Liquid/gas Permeability Testing



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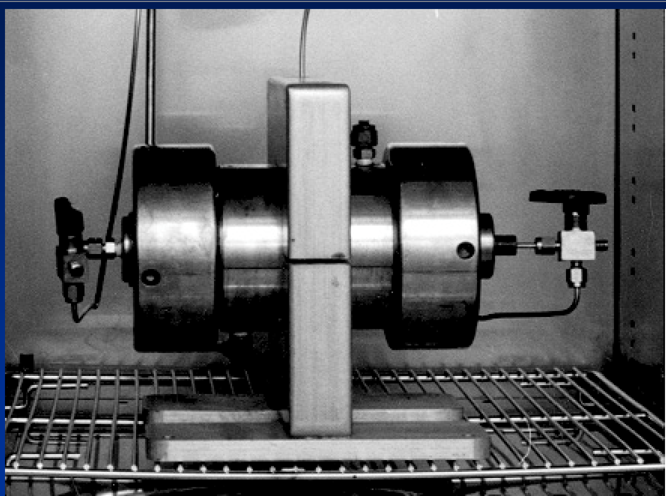
Reservoir Temperature Core Testing



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Reservoir Potential Damage Mechanism

- Natural fracture leakoff
- Fluid retention due to high capillarity
- Migratable fines
- HCl acid-sensitive minerals
- Mud acid sensitivity
- Water sensitivity
  - Due to expandable clays
  - Due to salinity shock (clay dispersion)
  - Due to aqueous imbibition in undersaturated gas reservoirs (decreases hydrocarbon relative permeability)

Wellbore/Reservoir Potential Damage Mechanism

- Inorganic scales
- Organic scales (deposits)
- Perforation compaction
- Polymer plugging
- Solids invasion (drilling, dirty fluids)
- Filtrate invasion (OBM, WBM, cement, frac fluids, high pH)
- Emulsions
- Clay swelling, migration
- Non-clay fines migration
- Wettability alteration
- Water retention (water blocks)
- Acid re-precipitation products
- Acid sludge

Stimulation Recommendations
Individual Wells

- Acid stimulation
- Hydraulic fracture treatment
  - Acid-based
  - Water-based
  - Oil-based
  - Methanol-based
  - Gas-based
- Remedial treatments

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Field & Formation Studies

Yegua	Selma Chalk	Frontier
Redfork	Morrow (Texas Panhandle)	Oriskany
Hosston	Morrow (Permian)	Spiro
Almond	North Sea Chalks	Vicksburg
Dakota	Offshore Miocene Sands	Devonian

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Technology Transfer
Technical Service Reports
CD-ROM Update (1990-2004)

Slide 47

Lab Tours

- Geological Laboratories
  - Geological Services (2nd floor)
  - Special Core Analysis (2nd floor)
  - Geomechanics (1st floor)

Slide 48

Solids Analysis

- Objectives
  - #1 - Take care of the Customer
    - Internal (BJ)
    - External (operators)
  - Identify the cause of the problem.
  - Recommend an acceptable, cost-effective solution.
  - Recommend a cost-effective, preventative measure.

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Tools

- Stereomicroscope (20X preferable)
- Magnet
- Hotplate
- Torch / burner
- Centrifuge
- Separatory Funnel
- Miscellaneous glassware
- Acids (HCl, HCl/HF, etc)
- Solvents (water, xylene, acetone, hexane)

Slide 50

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BJ Services Chemicals

- Surfactants
- De-emulsifiers
- Mutual Solvents

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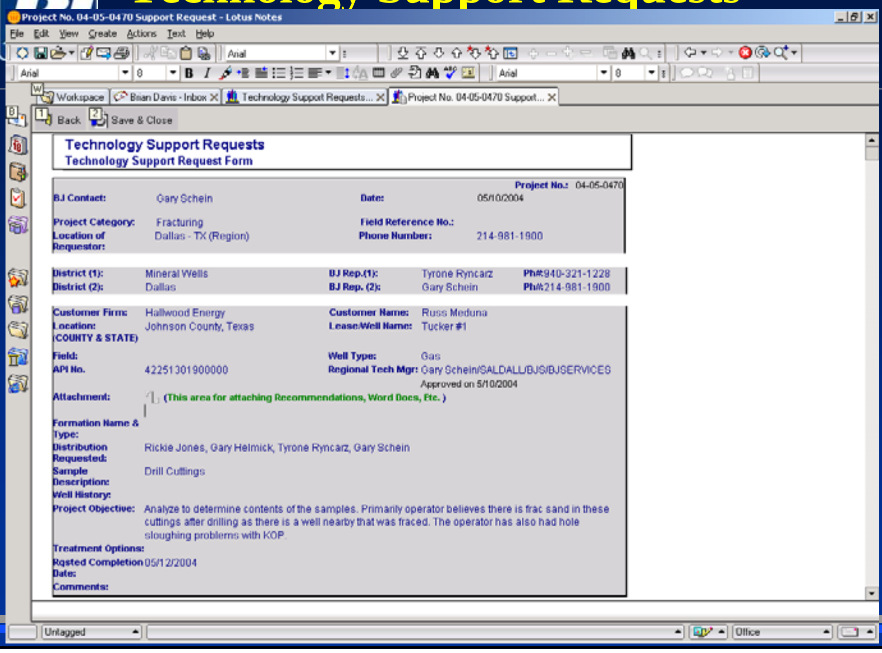
Technology Support Requests


Project No. 04-05-0470 Support Request - Lotus Notes

File Edit View Create Actions Text Help

Workspace | Brian Davis - Inbox | Technology Support Requests... | Project No. 04-05-0470 Support...

Back | Save & Close

Technology Support Requests
Technology Support Request Form

Project No.: 04-05-0470

BJ Contact:	Gary Schein	**Date:**	05/10/2004	
Project Category:	Fracturing	**Field Reference No.:**		
Location of Requestor:	Dallas - TX (Region)	**Phone Number:**	214-981-1900	
District (1):	Mineral Wells	**BJ Rep.(1):**	Tyrone Ryncarz	**Ph#**940-321-1228
District (2):	Dallas	**BJ Rep. (2):**	Gary Schein	**Ph#**214-981-1900
Customer Firm:	Hallwood Energy	**Customer Name:**	Russ Meduna	
Location: (COUNTY & STATE)	Johnson County, Texas	**Lease/Well Name:**	Tucker #1	
Field:		**Well Type:**	Gas	
API No.	42251301900000	**Regional Tech Mgr:**	Gary Schein/SALDALL/BJS/BJSERVICES Approved on 5/10/2004	

Attachment: (This area for attaching Recommendations, Word Docs, Etc.)

Formation Name & Type:

Distribution Requested: Rickie Jones, Gary Helmick, Tyrone Ryncarz, Gary Schein

Sample Description: Drill Cuttings

Well History:

Project Objective: Analyze to determine contents of the samples. Primarily operator believes there is frac sand in these cuttings after drilling as there is a well nearby that was fraced. The operator has also had hole sloughing problems with KOP.

Treatment Options:

Rqsted Completion Date: 05/12/2004

Comments:

Untagged | Office

Ask Questions!!

- Origin of sample?
  - Obtained how?
    - Flowed back, bailed, scraped off pump, etc.
- Well History?
  - New / Old completion?
  - Prior stimulation or remedial treatment?
- Type of well?
  - Producer
  - Injector (waterflood or salt water disposal).
- Production?
  - Oil, gas, water, or combinations?

Slide 53

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Solids Types
Organic Solids

- Paraffins
- Asphaltenes
- Paint chips
- Rubber packers, etc.
- Fluid loss additives

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Solids Types

Combinations - Organic + Inorganic

- Emulsions (oil, water, inorganics)
- Pipe dope (grease, inorganics)

Solids Analysis

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Solids Types
Inorganic

- Wellbore scales
  - Carbonates, iron oxides/hydroxides, sulfates, sulfides, etc.
- Proppants
- Formation materials
  - Sand, shale, fines, etc.
- Cement phases
- Drilling mud
- Phosphates
- Salts
- Perforating debris
- Amorphous solids

Inorganic Solids

- Tubing Scale

Inorganic Solids

- Tubing Scale

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Geological Laboratories Deliverables

- Single-well formation evaluation reports
- Solids analyses reports
- Field and formation studies
- Geomechanics Studies
- Coreflow studies
- Consulting and presentations
- Training

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ACID TESTING

INFORMATION FOR AE TRAINING

ACID TESTING

- BJ Acid Systems
- Quality Control of Acids
- Acid Problems
  - Emulsions
  - Acid sludges
  - Compatibility issues
- Corrosion Testing
- Corrosion Rates
- BJ Acid Corrosion Inhibitors
- Formation Reactions

ACID TESTING
BJ Acid Systems

- Mineral Acids
  - HCl
    - Used on formation >20% limestone & dolomite
    - Range from 28% down to 3% HCl
    - 28% HCl is legal transport limit
  - HCl+HF (aka Mud Acid)
    - Used on sandstone formations < 20% carbonates
    - 12% HCl+3%HF down to 6%HCl+1.5%HF
    - In certain cases down to 0.5% HF
    - Made by combining HCl with ABF or AF

ACID TESTING
BJ Acid Systems

- Organic Acids
  - Used in high-temperature wells (+300 ^{o}F).
    - Acetic (10-15%)
      - $(CH_3COO)_2Ca$ precipitation problems above 15% acetic acid
    - Formic (9-10%)
      - $(HCOO)_2Ca$ precipitation problems above 10% formic acid
    - Mixed (Organic + HCl and/or HF)

ACID TESTING
BJ Acid Systems

- Specialty Acids
  - Emulsified Acid
    - 30% diesel + 70% acid (15% HCl)
  - DeepSpot Acid
    - Crosslinked with XLA-2
  - BJ Sandstone Acid (BJSSA)
  - S3 (aka S-Cubed)
    - Scale/Iron control acid
  - Enhanced Acid
    - Self-diverting
  - StayLive Acid
    - Coats formation to slow reaction

ACID TESTING
BJ Acid Systems

- Specialty Acids
  - Sulfamic acid
    - Solid
    - “Acid Sticks”
    - Produces multiply H^+
  - Citric acid
    - Solid
    - Produces multiply H^+

ACID TESTING
Quality Control

- Acid Strength
  - Specific Gravity
  - Baume' Scale
  - Titration
- Acid Impurities
  - Iron
  - Solids
  - Color
  - Odor
- Special Cases

ACID TESTING
Quality Control

- Specific Gravity
  - Used to determine amount of HCl present.
    - S.G. of pure water = 1
    - Water + Acid = 1+x
    - Note: As temperature increases water S.G. decreases 0.001 for every 5 ^{o}F above 60 ^{o}F
  - Use correlation tables in Engineering Handbook to convert S.G. to % acid

ACID TESTING
Quality Control

- High temperature specific gravity correction.
  - As temperature increase, density of solution decreases which decreases specific gravity.
  - Example calculation
    - S.G. reading of 1.071 and temperature is 110 ºF.
      - 110 - 60 = 50
      - 50 / 5 = 10 (every 5 ºF over 60 ºF S.G. drops 0.001)
      - 10 x 0.001 = 0.01
      - 1.071 + 0.01 = 1.081
      - From Engineering Handbook: S.G. 1.081 = ~16.3% HCl

ACID TESTING
Quality Control

- Low temperature specific gravity correction.
  - As temperature drops, density of solution decreases which decreases specific gravity.
  - Example calculation
    - S.G. reading of 1.071 and temperature is 45 ºF.
      - 60 - 45 = 15
      - 15 / 5 = 3 (every 5 ºF under 60 ºF S.G. increases 0.001)
      - 3 x 0.001 = 0.003
      - 1.071 - 0.003 = 1.068
      - From Engineering Handbook: S.G. 1.068 = ~13.6% HCl

ACID TESTING
Quality Control

- Baume' Scale (°Bé)
  - Invented by French Chemist Antoine Baumé
  - Strength scale used by acid manufacturers.
  - Does not directly measure acid concentration.
  - To convert from °Bé to Specific Gravity at 60 ^{o}F: S.G. = 145/(145 - °Bé)

ACID TESTING
Quality Control

- Titration
  - Measures normality (N) of HCl
    - N = # gram equivalent weights of a solute per liter of solution
  - Sodium Hydroxide Solution
    - Either 0.2 or 2.0 N solution
  - Phenolphtalein Indicator
    - Light pink color for 30 seconds
  - $N_{HCl} = (N_{NaOH} * \Delta\ mL_{NaOH}) / mL_{HCl}$
  - Use correlation tables in Engineering Handbook to convert N_{HCl} to % acid

ACID TESTING
Quality Control

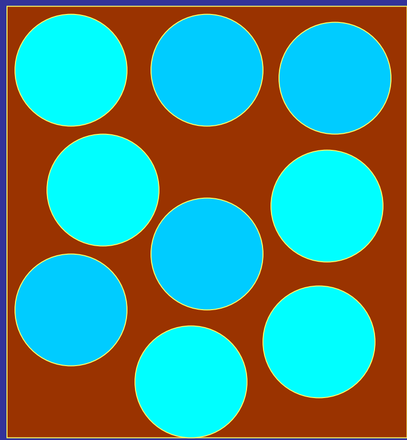
- Iron Content
  - Equal to or less than 100ppm in concentrated acid
- Solids
  - No solids
- Color
  - Yellow/green means Fe^{2+} or chrome
  - Orange means Fe^{3+}
- Odor
  - No sulfides

ACID TESTING
Quality Control

- Special Cases
  - Sulfides
    - Can interfere with corrosion inhibitors
  - Fluorides/sulfates
    - Interfere with Deep Spot Acid crosslinking
  - Organic acids
    - Titrate carefully
  - HF systems
    - Can interfere with corrosion inhibitors

ACID TESTING
Problem: Emulsions

- **Definition**
  - **Stable, viscous mixture of oil and water**
    - **Oil external**
    - **Water external**
- **Problem Areas**
  - **Gas wells? No**
  - **Oil wells? Yes**

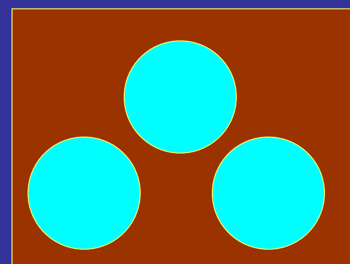


ACID TESTING
Problem: Emulsions

- **Do You Have An Emulsion?**
  - **Check specific gravity of production fluid**
    - **If low, then emulsion is present.**
  - **Water test to determine emulsion type**
    - **Oil external**
    - **Water external**
    - **Stabilized by fines or chemical films?**
  - **Determine % water**
    - **Centrifuge with emulsion breaker**

ACID TESTING
Problem: Emulsions

- **Preventative Action**
  - **Treat external phase**
    - **NE-940**
    - **NE-118**
      - **Change surface tension**
        - **Allows easier merging of droplets**
        - **Gravity takes over**
  - **Want 90% broken in 15 minutes**
    - **Mix**
    - **Heat**
    - **Screen**



ACID TESTING
Problem: Sludges

- **Definition**
  - **Asphaltene precipitation**
- **Testing**
  - **Pre-screen oil**
  - **Heat set time (0.5 to 4+ hours @ 150 F)**
  - **Re-screen**
  - **Retest with iron added**
    - **1250 Fe^{3+} + 3750 Fe^{2+} = 5000ppm total Fe**
  - **Evaluate additives**
- **Interpret Results**
  - **< 0.05g / 50 mL crude oil (visible limit)**

ACID TESTING
Problem: Sludges

- TYPES OF IRON
  - Ferric
    - Fe^{3+}
    - Most oxidized
      - rust
      - forms in oxygenated conditions
    - Mainly comes from rusted tubulars
    - Responsible for most sludges
      - crosslinks asphaltenes
    - Ferric hydroxide gel precipitates starting at ~ pH 2.2 and increases as pH increases

ACID TESTING
Problem: Sludges

- TYPES OF IRON
  - Ferrous
    - Fe^{2+}
    - Less oxidized
    - Comes from formation and acid attack on steel
    - Less troublesome
    - Ferrous hydroxide precipitates at pH 7.7
    - Will convert to Fe^{3+} when oxygen is present

ACID TESTING
Problem: Sludges

- Corrective Action
  - Pre-flush
    - HCl/KCl/surfactants/mutual solvent/xylene
    - Removes oil from well bore
  - Pickling
    - Removes rust (Fe^{3+})
    - Keep acid out of the formation
    - Don't pickle chrome steels!
      - They don't rust.
      - Very susceptible to acid attack.
  - Dissolve with Xylene
    - Either preflush or mixed with acid

ACID TESTING
Problem: Sludges

- Corrective Action
  - Reducing agents
    - Convert Fe^{3+} to Fe^{2+}
    - Don't work well if H_2S present
  - Chelating agents
    - Ties up iron
    - Don't work well below pH 3
    - Good for gas wells

ACID TESTING
Problem: Compatibility

- Solubility of Additives
  - Oiling out
    - Mutual Solvent
    - Xylene
    - Diesel
- Reaction with Additives
  - Cationic inhibitors + anionic surfactants

ACID TESTING
Corrosion Testing

- Low Pressure Corrosion Tests
  - Water bath
    - 185 ^{o}F
    - Atmospheric pressure
- High Pressure Corrosion Tests
  - Chandler Corrosion Apparatus
    - 500 ^{o}F
    - 5000 psig

ACID TESTING
Corrosion Rates

- Corrosion Rates
  - Coiled tubing
    - 0.02 lbs/ft^2
    - 0 on pitting scale (0 - 5)
  - Other tubulars
    - 0.05 lbs/ft^2
    - 0 - 2 on pitting scale (0 - 5)

ACID TESTING
Acid Corrosion Inhibitors

- VERY TOXIC!!!!!!!
- Perform better at pressure increases.
- Determining loadings concentration
  - Acid type
    - HCl, mud, or organic acid?
  - Temperature
    - Above or below 250 ^oF
  - Metals present
    - Carbon, chrome, or coiled tubing?
  - Contact time

ACID TESTING
Acid Corrosion Inhibitors

- Acid type
  - Organic acid
    - CI-11, CI-28, CI-29, CI-31, CI-33
  - Mineral acid
    - CI-25, CI-26, CI-27, CI-29, CI-30, CI-31

ACID TESTING
Acid Corrosion Inhibitors

- Locations
  - USA
    - CI-11, CI-25, CI-27, CI-31
  - North Sea
    - CI-26, CI-29
  - Europe/Africa
    - CI-11, CI-25, CI-27, CI-30
  - Middle East
    - CI-25
  - Asia/Pacific
    - CI-25, CI-30
  - South America
    - CI-25, CI-30, CI-31

ACID TESTING
Formation Testing

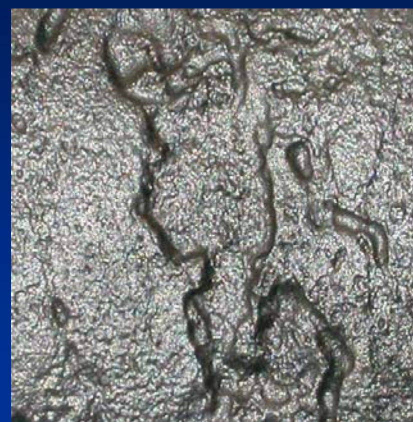
- Formation Solubility
  - Measured
    - Gravimetrically
    - Carbon dioxide production
  - Acid picks
    - HCl on limestone
    - Mud / BJSSA (HCl:HF) on sandstone and clays
    - Organics in high temperature wells

ACID TESTING

- Questions?

One of a Twelve:
Picking the Right Acid Corrosion Inhibitor

- Tools to Prevent:
  - Damaged Equipment
  - Ruined Wells
  - Loss of Life



Slide 1

Who Am I?

- Mark Vorderbruggen
  - Ph.D. Chemist
  - NACE Metallurgist
  - 10 Years Experience
  - Patented Chemistry
  - Invented CI-29, CI-31
  - Approval Tester for CI-33, CI-34, HTACI

Slide 2

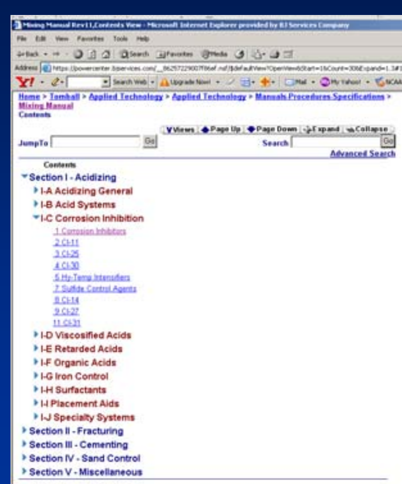
One of Twelve: Why So Many ACI's?

- Chemical Reasons
  - Acids
  - Metals
  - Temperatures
  - Additives
- Non-Chemical Reasons
  - Economics
  - Inventory
  - Environmental Laws

Slide 3

Tool 1: Mixing Manual

- Overview of ACI's
  - Compatibilities
  - Temperature Limits
  - Suggested Loadings
- Benefits
  - Easy Access
  - Looks Good
  - Prevents Simple Mistakes



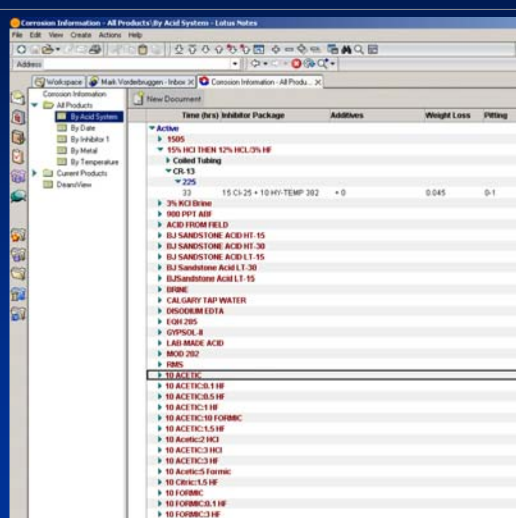
Slide 4

Tool 2: Acid Database

- Lotus Notes Database
- Sum Of All Knowledge
  - 33,000 Tests
  - Full Details
  - Some Competitor ACI's
- Searchable
- Works/Doesn't Work

Slide 5

Tool 2: Acid Database



Slide 6

Tool 3: Acid Lab

- Personalised Service
  - Experience
  - Current Results
  - Testing
    - HPHT Autoclaves
    - Metal Library
    - Additives

Slide 7

Tool 3: Acid Lab

- Test Procedure
  - Design Plan
  - Run Test
  - Analyze Results
  - Optimize Loadings
  - Submit Report

Slide 8

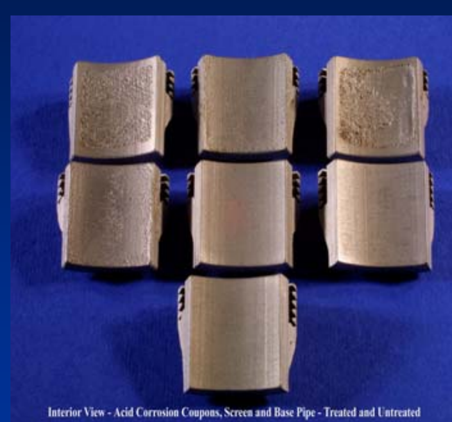
Conclusions

- Almost Infinite Combinations
  - Acids
  - Metals
  - Temperatures
- Wrong Pick Leads to Disaster
  - Damaged Equipment
  - Ruined Wells
  - Loss of Life

Slide 9

Conclusions

- Tools to Help You
  - Mixing Manual
    - Fast
  - Acid Database
    - Detailed
  - Acid Lab
    - Optimized



Interior View - Acid Corrosion Coupons, Screen and Base Pipe - Treated and Untreated

Slide 10

Slide 11

Acid Corrosion Inhibitors

by Mark A. Vorderbruggen, Ph.D.

Introduction

- Definitions
- Electrochemistry
- Acid Attack on Steel
- Inhibitor Mechanisms
- Inhibitor Chemistry
- Other Factors
- Intensifier Chemistry
- Summary

Corrosion: A Definition

- Latin “Corrous”
- Gradual Destruction
- Extensive Reactions
- Electrochemical Processes

Corrosion Definitions

- Uniform Corrosion
  - Generalized
- Pitting Corrosion
  - Localized
- Stress Corrosion
  - Under pressure
- Hydrogen Embrittlement
  - H_2 in the lattice

Corrosion Definitions

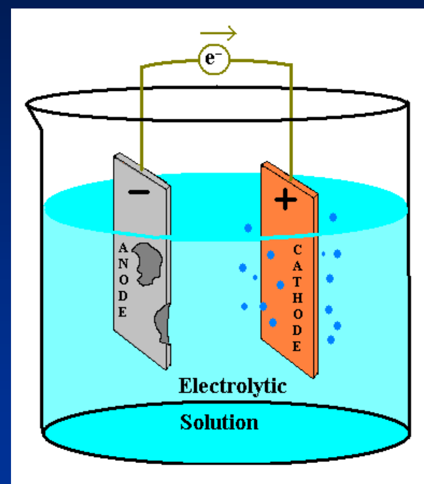
- Galvanic Corrosion
  - Oxidation-reduction process
  - Dissimilar electron distribution
- Anode
  - Low electron density
  - Attracts anions
  - Suffers corrosion
- Cathode
  - High electron density
  - Attracts cations
  - Remains untouched

Corrosion Definitions

- Mechanism Of Process
  - Converting reactants to products
- Corrosion Rate
  - Rate of metal loss over an exposure time
  - Pounds per square foot of tubular surface
    - lbs/ft^2
  - Millimeters per year
    - mpy
    - To convert lb/ft^2 to mpy, multiply lb/ft^2 by 628

Electrochemistry

- Moving Electrons
  - Anode
    - Attracts anions (Cl^-, OH^-)
    - Loses electrons
    - Corrodes
    - "-" terminal of battery
  - Cathode
    - Attracts cations (H^+)
    - Gains electrons
    - Remains intact
    - "+" terminal of battery



Revised 01/13/2004 Slide 7

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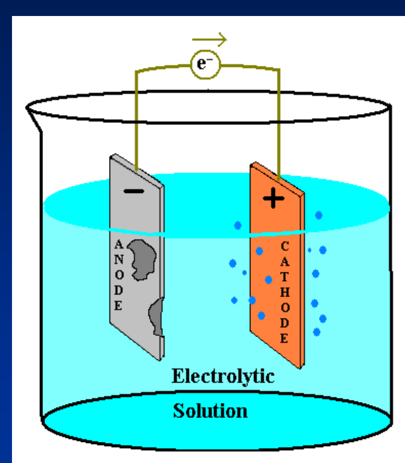
Electrochemistry

- Half-Cell Reactions
  - Zinc/Copper in acid
    - Oxidation of zinc at anode
      $Zn^o \longrightarrow Zn^{2+} + 2e^-$
    - Reduction of oxygen at copper cathode
      $O_2 + 4H^+ + 4e^- \longrightarrow 2H_2O$



Revised 01/13/2004 Slide 8

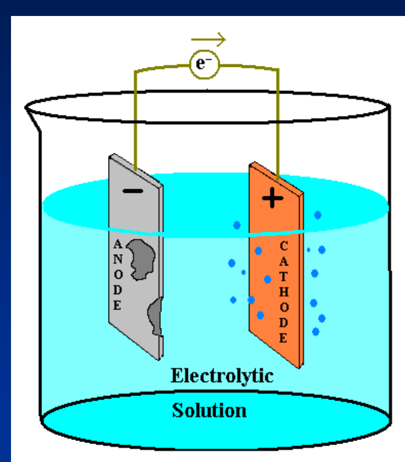
EDC, Tomball, TX

Electrochemistry

- Corrosion = Battery
  - Two dissimilar metals in contact
  - Electrolytic solution
  - Moving electrons

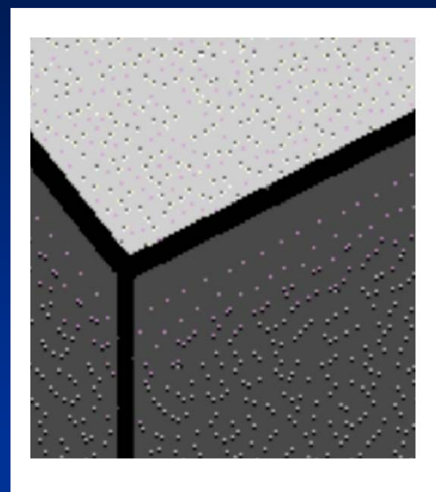
Electrochemistry

- Moving Electrons
  - Galvanic series
    - Platinum
    - Gold
    - Silver
    - Copper
    - Brass
    - Lead
    - Chrome steel
    - Aluminum
    - Zinc
    - Magnesium



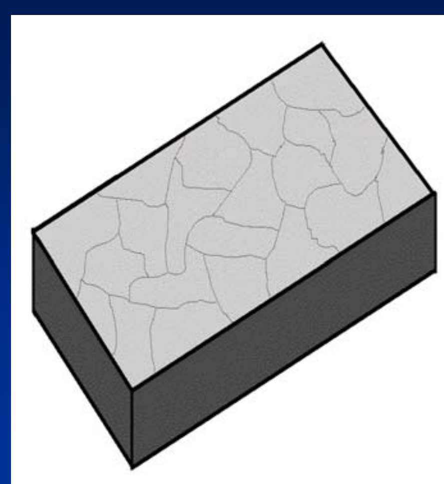
Acid Attack On Steel

- Structure of Metals
  - Atomic level
    - Nuclei suspended in a fog of electrons.
    - Easy flow of electrons.



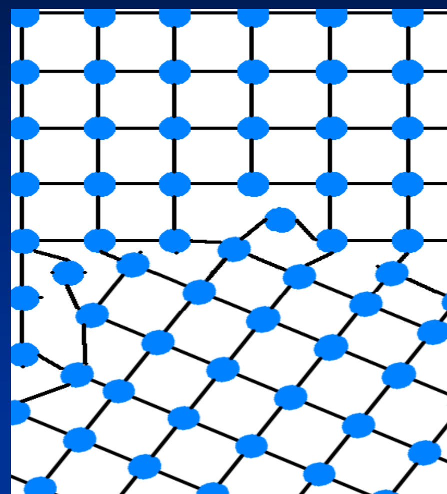
Acid Attack On Steel

- Structure of Metals
  - Microscopic level lattice disturbances
    - Grain boundaries
    - Phase differences
    - Inclusions
    - Damaged areas



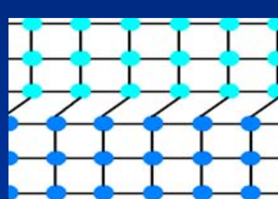
Acid Attack On Steel

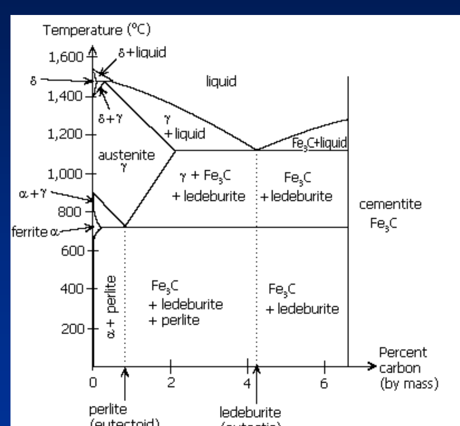
- Lattice disturbances
  - Grain boundaries
  - Formed as metal solidifies
  - Benefits?
    - Many small grains give strength and toughness



Acid Attack On Steel

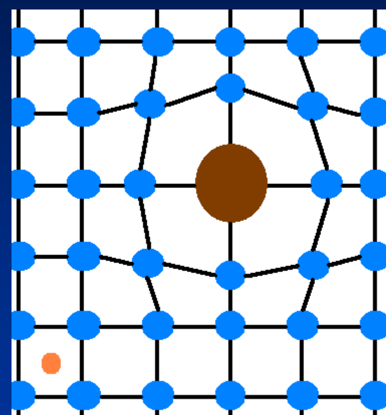
- Lattice disturbances
  - Phase differences
    - Austinite (FCC)
    - Ferrite (BCC)
    - Martensite (DBCC)
    - Affect hardenability





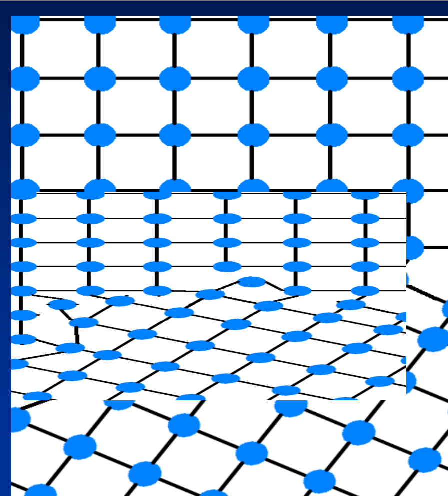
Acid Attack On Steel

- Lattice disturbances
  - Inclusions
    - Alloying gives enhanced properties
    - Contaminants harm desired properties



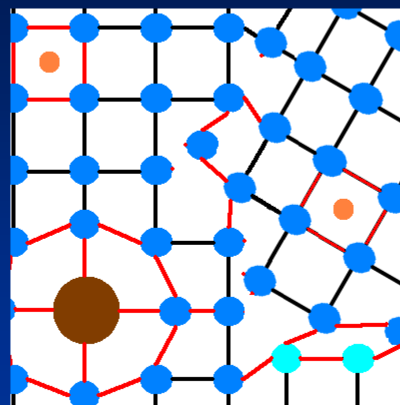
Acid Attack On Steel

- Lattice disturbances
  - Damaged Areas
    - Mechanical stress
      - Cold working
      - Tool damage
        - Crystal structure becomes deformed
        - Metal hardness increases



Acid Attack On Steel

- Structure of Metals
  - Lattice disturbances
    - High energy



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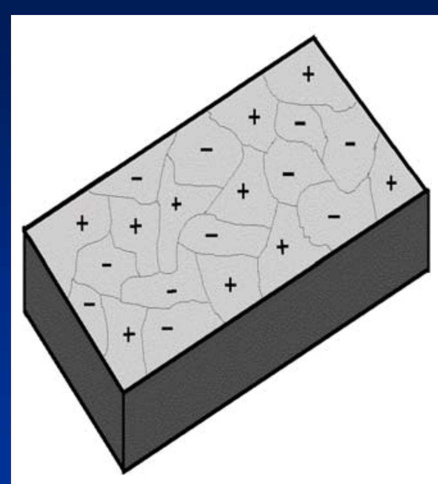
EDC, Tomball, TX

Acid Attack On Steel

- Structure of metals
  - Anodic/Cathodic areas develop.
    - Anodes attract anions (-)
    - Cathodes attract cations(+)
  - No longer need dissimilar metals for galvanic corrosion.



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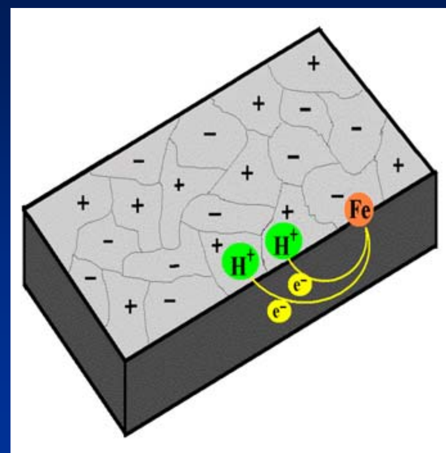
EDC, Tomball, TX

Acid Attack On Steel

- Corrosion occurs
  - Hydrogen cations attracted to cathodic regions.
  - Electrons transfer from iron “electron fog” to two hydrogen ions.
  - Half-cell reactions:
    - Oxidation at anode
    - $Fe^{o} \longrightarrow Fe^{2+} + 2e^{-}$
    - Reduction at cathode
    - $2H^{+} + 2e^{-} \longrightarrow H_2$



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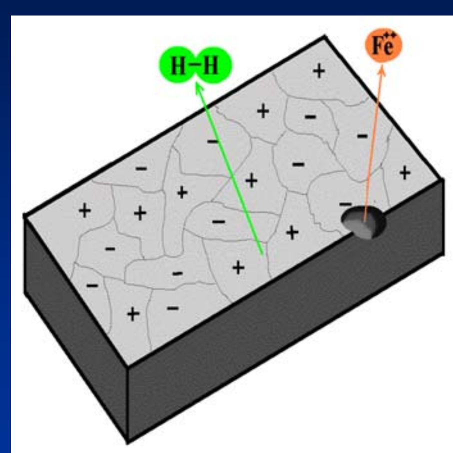
EDC, Tomball, TX

Acid Attack On Steel

- Corrosion occurs
  - Electrons transfer from one iron atom to two hydrogen ions.
    - Iron atom oxidized to ferrous ion.
    - Two hydrogen ions are reduced to hydrogen gas
  - Ferrous iron cation and hydrogen gas flow/pulled away from surface.



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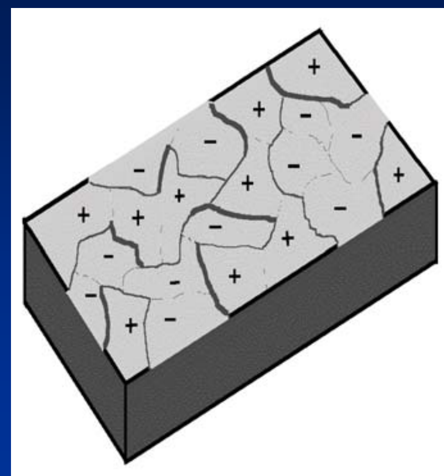
EDC, Tomball, TX

Acid Attack On Steel

- Corrosion occurs
  - Loss of iron results in pitting at anodic sites of steel's surface.



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EDC, Tomball, TX

Inhibitor Mechanisms

- Inhibitor types
  - Cathodic inhibitors
  - Anodic inhibitors
  - Mixed inhibitors

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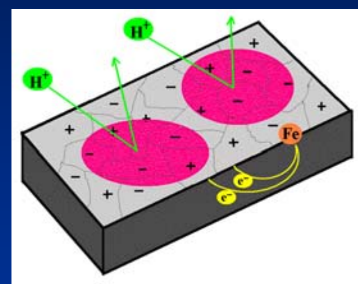
EDC, Tomball, TX

Inhibitor Mechanisms

- Inhibitor types
  - Cathodic inhibitors
    - Stops hydrogen ions from reaching surface.
    - Prevents hydrogen half-cell reduction reaction.
    - $2H^+ + 2e^- \longrightarrow H_2$



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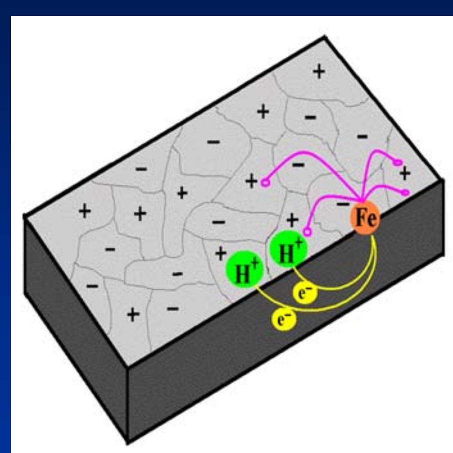
EDC, Tomball, TX

Inhibitor Mechanisms

- Inhibitor types
  - Anodic inhibitors
    - Stops Fe^{2+} from escaping.
    - Fe^{2+} reabsorbs electrons.
    - Prevents iron half-cell oxidation reaction.
    - $Fe^o \longrightarrow Fe^{2+} + 2e^-$
    - Passivation is an anodic inhibitor



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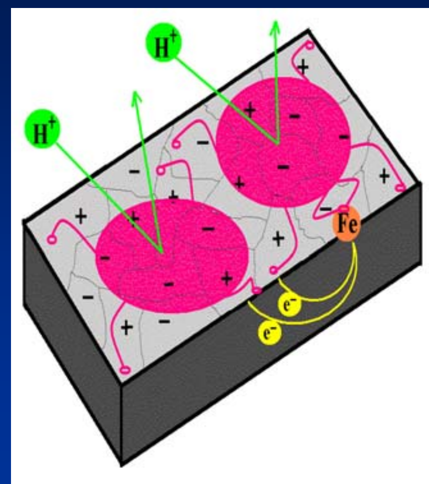
EDC, Tomball, TX

Inhibitor Mechanisms

- Inhibitor Mechanisms
  - Mixed inhibitors
    - Blocks both anodic and cathodic reactions.



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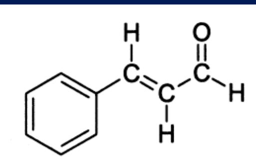
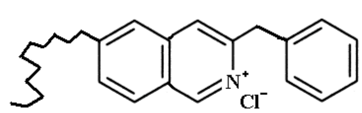
Inhibitor Chemistry

- General features
  - Electronegative atoms
    - Nitrogen
    - Oxygen
    - Sulfur
  - Loosely held electrons
    - π-bonds (double bonds)
    - Aromatic rings
  - Planer structure

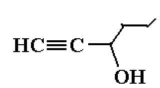
  - Create a hydrophobic zone at metal's surface



Cinnamaldehyde



Nitrogen Quat

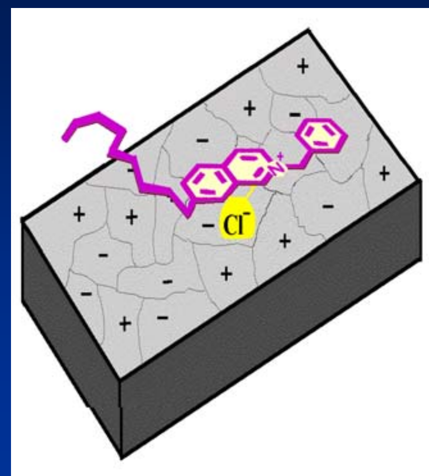


Propargyl Alcohol

Revised 01/13/2004 *Slide 26*

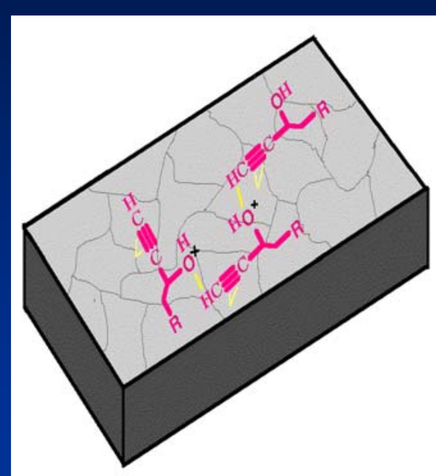
Inhibitor Chemistry

- **Primary**
  - **Inhibitor molecules bind to metal "as is".**



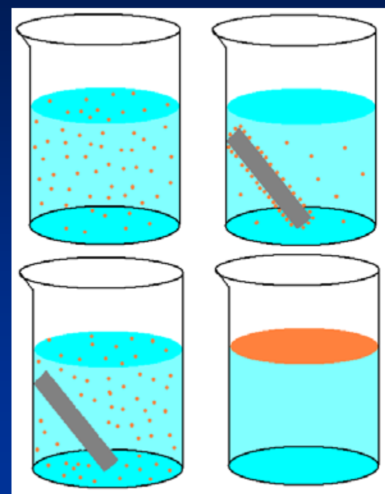
Inhibitor Chemistry

- **Secondary**
  - **Inhibitor molecules undergo chemical change resulting in improved inhibition.**



Other Factors

- **Surfactants**
  - **Assist in dispersing inhibitor molecules in acid.**
    - **To much keeps inhibitor in acid**
    - **To little lets inhibitor "oil out" of acid**
- **Solvents**
  - **Tie inhibitor package together.**



Other Factors

- **HCl**
- **HCl + HF**
- **Organic**
- **Speciality**
  - Emulsified Acid
  - StayLive Acid
  - DeepSpot Acid
  - BJ Sandstone Acid
  - S3 (aka S-Cubed)
  - Enhanced Acid
  - Divert S

Other Factors

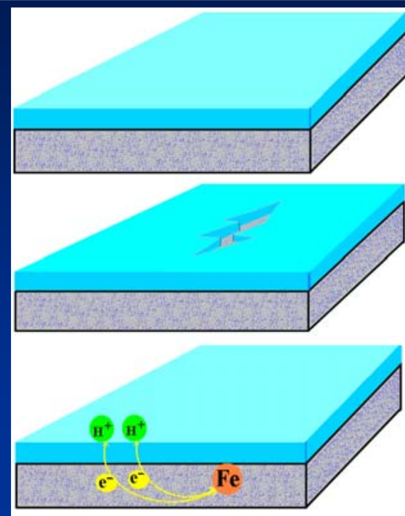
- Steel Issues
  - Different steels interact differently with inhibitors.
    - Alloys
    - Production

Other Factors

- Steel Grades & Alloys
  - Casing and tubing
    - J-55, K-55, L-80, N-80, C-90, C-95, T-95, P-110, Q-125
  - Drill pipe
    - E-75, X-95, G-105, S-135
  - Coiled tubing
    - QT-700, QT-800, QT-900, QT-1000, HS-80, HS-110
  - Tool steels
    - 4130, 4140, 4145, 8620
  - Low carbon
    - 1010, 1018

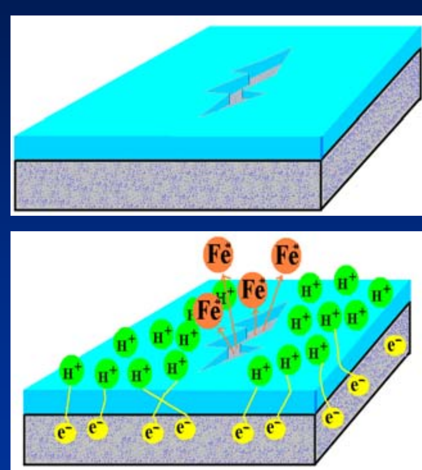
Other Factors

- Steel Alloys
  - Chrome steels
    - 304, 316, Cr11, Cr13, Cr2205, Cr2535, Cr2707, Cr25, Nitronic-30, Super Chrome13, Super Chrome13-Modified
  - Chrome problem
    - Oxide layer passivation
    - Anodic inhibitor
    - Prevents loss of Fe^{2+}



Other Factors

- Steel Issues
  - Chrome problems continued
    - HCl removes chrome from oxide layer.
    - Holes appear in oxide layer.
    - Fe^{2+} removal occurs through holes.
      - Cathode surface is relatively huge
      - Anode surface localized at holes
      - Result: fast, focused loss of iron



Other Factors

- Steel Issues
  - Production
    - Same alloys can be worked different ways.
      - American vs. Japanese
      - Heat treatments
      - Cold working
      - Welding
    - Results
      - Different grain structures.

Other Factors

- Phosphates, Silicates and Organic Sulfonates
  - May precipitate inhibitors
- Monovalent Cations
  - No measurable effect
- Divalent Cations
  - High concentrations interfere

Other Factors

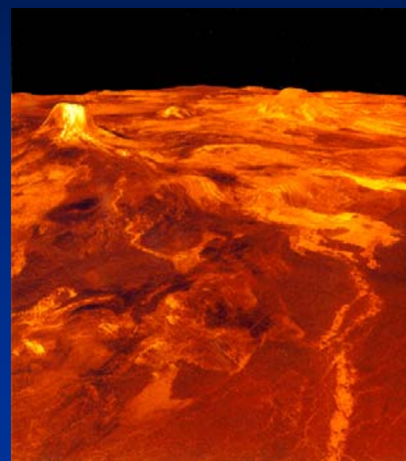
- Chloride Anion
  - Strongly adsorbed by steel
  - Make it difficult to passivate (anodic inhibition)
  - More passivating inhibitor required with high chlorides
    - Passivating inhibitors are NOT used in HCl
- Sulfate Anion
  - May cause clumping of certain long carbon chain corrosion inhibitors

Other Factors

- Sulfides
  - Reduced to free sulfur by oxidizing inhibitors
- Naphthenic acids (R-COOH)
  - Require extra inhibition in acidic environments
- Oxygen (Rust)
  - Organic inhibitors not generally effective
  - Must contain passivating groups such as benzoates and sulfonates

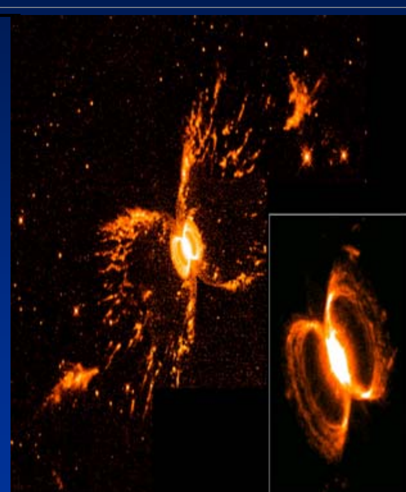
Intensifiers

- Intensifier Properties
  - Used
    - High temperatures
    - Strong acid strengths
    - Long jobs
  - Do not function as inhibitors by themselves.
  - Assist inhibitors
    - Lessen pitting
    - Increase duration of inhibitor protection



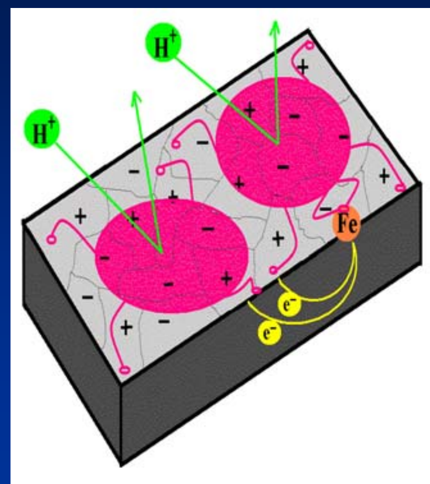
Intensifiers

- Common intensifiers
  - Bridges
    - Formic acid
      - $HCOOH \rightarrow CO$
    - Iodine
      - $I_2 \rightarrow I^+ + I^-$
      - Binds to aromatics
    - Copper salts
      - Binds to alkynes
      - Especially good with Cr steels
  - Passivators
    - Antimony salts
    - Mercury salts



Summary

- What slows corrosion?
  - Interfering with reactions
    - Oxidation at anode
    - $Fe^{o} \longrightarrow Fe^{2+} + 2e^{-}$
    - Reduction at cathode
    - $2H^{+} + 2e^{-} \longrightarrow H_2$



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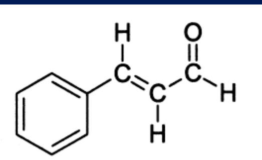
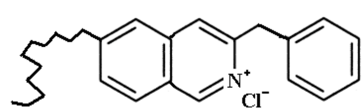
Summary

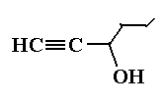
- How?
  - Molecules containing
    - Electronegative atoms
    - Loosely held electrons
    - Planer structure



Cinnamaldehyde



Nitrogen Quat



Propargyl Alcohol

Revised 01/13/2004 Slide 42

EDC, Tomball, TX

Summary

- What else?
  - Surfactants
  - Solvents
  - Knowing your steel

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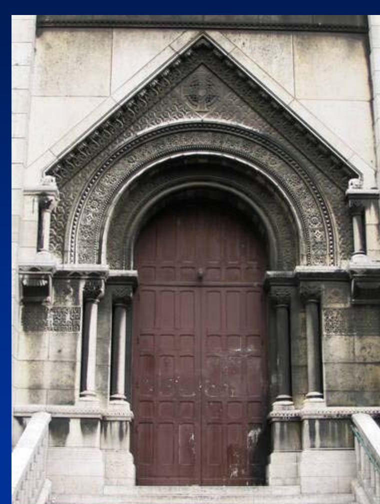
EDC, Tomball, TX

Acid Corrosion Inhibitors

- Thank You!
  - Original graphics by Mark Vorderbruggen
  - Photographs supplied by
    - http://www.freeimages.co.uk/
    - http://www.mayang.com/textures/index.htm



Revised 01/13/2004

EDC, Tomball, TX

Fracturing Fluids

Fluid Objectives

1. Create the Fracture (initiate and propagate)
2. Carry the Proppant
3. Suspend the Proppant
4. Minimize Damage
5. Safe, Easy to Use, Green

Fluid Properties

1. High Viscosity
2. Reservoir Compatibility
3. Must Break to Low Viscosity
4. Temperature Stability

Water Based Fracturing Fluids

- **Essential Ingredients**
  - **High Water Quality**
  - **Water Soluble Polymer**
  - **Buffer**
  - **Crosslinker**
  - **Breaker**

FAMILY OF FRACTURING FLUIDS

Temperature °F: 50° 75° 100° 125° 150° 175° 200° 225° 250° 275° 300° 325° 350° 375° 400°
Temperature °C: 10° 24° 38° 52° 66° 79° 93° 107° 121° 135° 149° 163° 177° 190° 204°

Zirconium-Crosslinked Fluids	
Vistar	50°F to 400°F - 10°C to 149°C
Low PH Vistar	50°F to 250°F - 10°C to 149°C
Medallion Frac	60°F to 275°F - 16°C to 135°C
Medallion Frac HT	200°F to 400°F - 93°C to 204°C
Borate-Crosslinked Fluids	
Spectra Frac G	50°F to 330°F - 10°C to 165°C
Viking	60°F to 175°F - 16°C to 79°C
Viking D	80°F to 300°F - 27°C to 149°C
Lightning	50°F to 300°F - 10°C to 149°C
Green Lightning	50°F to 300°F - 10°C to 149°C
Non-Aqueous Fluids	
Super Rheo-Gel	50°F to 325°F - 10°C to 121°C
Methofrac	50°F to 300°F - 10°C to 104°C
Viscoelastic Surfactant Fluids	
AquaClear	50° to 150°F- 10° to 66°C
Elastra Frac	50°F to 250°F - 10°C to 121°C
Acid Fracturing Fluids	
Dual Phase Acid	50°F to 225°F - 10°C to 107°C
DeepSpot Acid	50°F to 300°F - 10°C to 177°C

Water Based Systems

Zirconium Crosslinked Fluids

- Vistar™ Fracturing Fluid (*)
- Patented low-polymer (CMG) system
  - Useful from 70°F to 400°F
- Vistar LpH™ Fracturing Fluid (*)

  Low pH, low-polymer (CMG) system
  - Useful from 60°F to 250°F
- Medallion™ Fracturing Fluid - Low pH CMHPG system
  - Useful from 60°F to 275°F
- Zirconium crosslinks at pH 3-6 and at 9-12
- Zirconium forms permanent bonds with polymer (covalent)

(*) Denotes proprietary, BJS patented technology

Borate Crosslinked Fluids

- Spectra Frac G™ Fracturing Fluid (*) Organoborate cross-linked guard system
  - Useful from 60° F to 330°F
- Viking™ Fracturing Fluid - Economic borate cross-linked guar system
  - Useful from 60°F to 175°F
- Viking D™ Fracturing Fluid - Delayed borate cross-linked guar system
  - Useful from 80°F to 300°F

(*) Denotes proprietary, BJS patented technology

FRACTURING FLUIDS

LIGHTNING

The ***Lightning*** system incorporates the use of a newly developed high yield guar polymer (GW-3) as a replacement to (GW-4) in all of the borate crosslinked fluids BJ Services provides, i.e. ; Viking, Viking D and Spectra Frac. The Lightning system has practical applications from 60 - 250°F BHST

- **High Yield allows for lower polymer loadings (15-25 ppt)**
- **Better tolerance to Mix Waters than Vistar & includes 2% KCl**
- **Can reduce polymer loading resulting in less residue & job cost**

SpectraStar

- **Organoborate-xlinked high yield guar system**
- **BHSTs from 60 - 300°F**
- **Incorporates BJ-proprietary SpectraFrac crosslinkers**
- **Polymer loadings reduced by up to 20% vs. SpectraFrac**
- **Internal breaker mechanism like SpectraFrac**
- **Less sensitive to water quality than Vistar systems**
- **Can be used with KCl**

Slickwater Fracs

- **FRW-14 friction reducer (2%KCl or produced waters)**
- **FRW-15 or FRW-15A friction reducer (fresh water)**
- **pumped at high rates**
- **low proppant concentrations or LiteProp**

Non-Aqueous Fluids

- Super RheoGel™ Fracturing Fluid
  Phosphate ester **based oil** gellation system
  - Useful from 80°F to 300°F
- MethoFrac XL™ Fracturing Fluid (*)
  Anhydrous **methanol** gellation system
  - Useful from 50°F to 220°F

(*) Denotes proprietary, BJS patented technology

Specialty Fluids

- Aqua Clear™ Fracturing Fluid (*) - Surfactant based gel system technology
  - Useful to 140°F
- Elastra Frac™ Fracturing Fluid (*) - Surfactant based gel system technology
  - Useful to 200+°F
- Aqua Frac™ Fracturing Fluid - Linear gelling system
  - Any polymer from our product line as required
- Polyemulsion - water/oil emulsion system
  - Useful to 200+°F

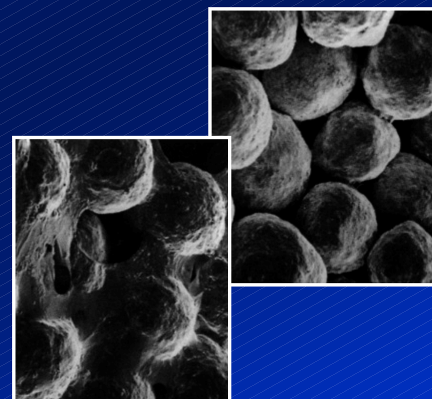
(*) Denotes proprietary, BJS patented technology

Breaker Technology

- Designed to produce predictable break performance over a wide range of formation parameters
- Specific to base fluid utilized
- Variable application methods
- Optimized for frac fluid clean-up to maximize retained conductivity

Water-Based Fluid Breakers

- Oxidizers
  - GBW-5, GBW-7, GBW-18, GBW-23, GBW-24
- Encapsulated Oxidizers
  - High Perm CRB, High Perm CRB LT, High Perm BR, High Perm KP
- Conventional Enzymes
  - GBW-10, GBW-12, GBW-15™ Breaker, GBW-33D™ Breaker



*White denotes proprietary, BJS patented technology

Water-Based Fluid Breakers

- Encapsulated Linkage Specific Enzyme -
  - High Perm CRE LpH™ Breaker
- Polymer Linkage Specific Enzymes -
  - EnZyme G™ Breaker (GBW-12CD),
  - EnZyme C™ Breaker (GBW-26C)
  - EnZyme S™ Breaker (GBW-16C)
  - EnZyme X™ Breaker
  - High Perm CRE™ Breaker

*White denotes proprietary, BJS patented technology

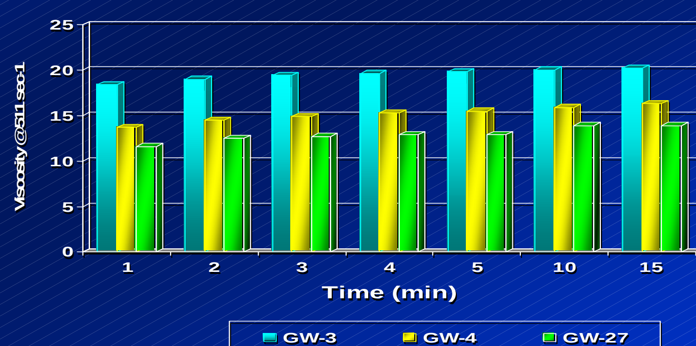
Oil-Based Fluid Breakers

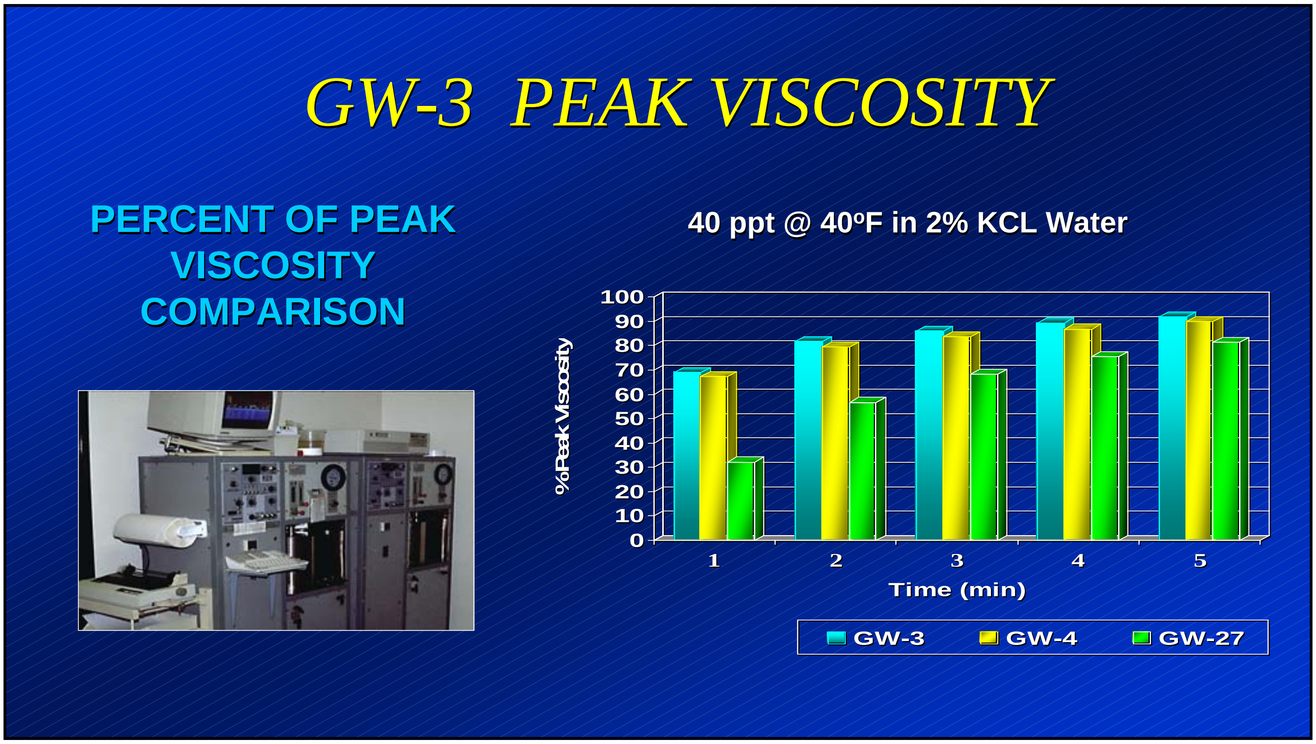
- Solid Breakers
  - GBO-6
- Liquid Breakers
  - GBO-5L, GBO-5LT, GBO-9L, GBO-9LT

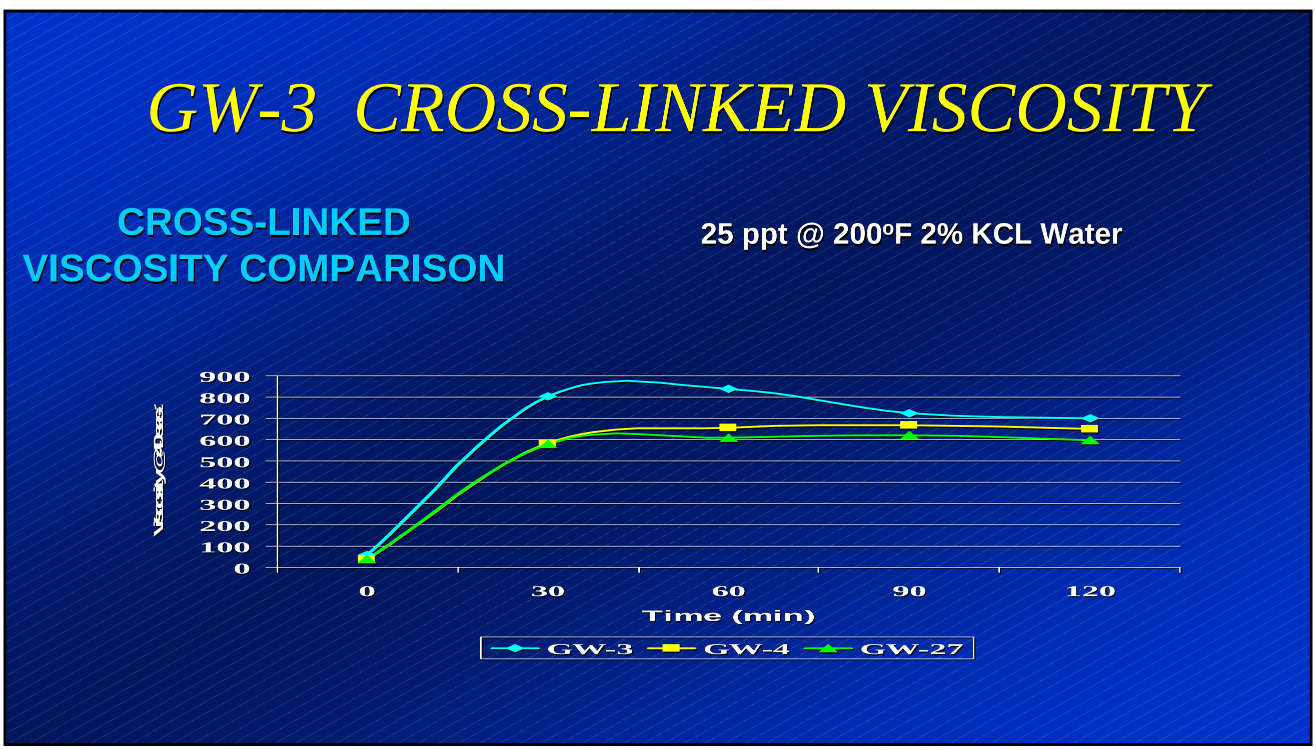
GW-3 HYDRATION

POLYMER HYDRATION COMPARISON

25 ppt Polymer @ 75ºF 2% KCL Water




GW-3 PEAK VISCOSITY
PERCENT OF PEAK VISCOSITY COMPARISON
40 ppt @ 40ºF in 2% KCL Water
% Peak Viscosity
100
90
80
70
60
50
40
30
20
10
0
1
2
3
4
5
Time (min)
GW-3
GW-4
GW-27


GW-3 CROSS-LINKED VISCOSITY
CROSS-LINKED VISCOSITY COMPARISON
25 ppt @ 200ºF 2% KCL Water
900
800
700
600
500
400
300
200
100
0
0
30
60
90
120
Time (min)
GW-3
GW-4
GW-27

Slurries Hydration Time

XLFC-1	1
XLFC-5	2
XLFC-3	3
VSP-1	4

#4 the fastest and the # 1 the slowest

Lab Tests

- ***Water based Fluids***:
  - ✓ Spectra Frac G
  - ✓ Viking
  - ✓ Vistar
  - ✓ ElastraFrac
- ***Oil based Fluid***:
  - ✓ Super Rheo Gel
- ***Methanol based Fluid***:
  - ✓ MethoFrac XL

Lab Equipment

- ***Linear gel Viscosity***

  FANN Model 35

  Computerized Viscometers

  OFITE M900

  Grace 3500

- ***Crosslinked Fluids***:

  FANN 50

  Grace 5500

Rheology

Apparent Viscosity

- Frac fluid properties evaluated by steady-shear rheological measurements
  - Fann 35 and 50 Viscometers (concentric cylinder)
  - RCV (Reciprocating Capillary Viscometer)
- Measure the apparent viscosity of the fluid as function of
  - shear rate
  - temperature
  - fluid composition
  - time

Apparent Viscosity

- determines friction pressure in the tubing and in the fracture
- determines ability of fluid to carry proppant
- determines fracture area
- determines fracture width
- affects fluid loss

Viscosity

- Viscosity is internal resistance a fluid has to flow due to the frictional force which arises when one layer of fluid moves across another
- Viscosity characterization involves measurements of stress that results from applying a known shear rate on the fluid.
- Fluid properties are described by the relationship between flow rate (shear rate) and the pressure (shear stress) that caused the movement
- Apparent viscosity dependent on the shear a fluid experiences at a specific point

Rheology

Shear Rate and Viscosity

- Apparent viscosity μ is ratio of shear stress to the shear rate defined as being equal to shear stress divided by shear rate as shown in the following equation. $\mu = \frac{\tau}{\gamma}$

Where

μ	=	Apparent Viscosity
τ	=	Shear Stress (Du/Dr)
γ	=	Shear Rate

Rheology

- Shear Stress τ is shearing force per unit of surface
  - measure torque exerted on measurement bob (Fann 50)
  - measure the pressure drop across a tube (RCV)
- Shear rate γ is velocity difference between the fluid layer divided by the distance between the fluid layer.
  - measure rpm of cup (Fann 50)
  - measure flow rate (RCV)

Shear Stress for Couette flows –

- Shear Stress for Couette flows –

$$\tau b = 3.344 \times 10^{-4} \frac{T}{Rb^2\,L},\ \frac{lbf}{ft^2}$$

- Where T (torque) = $k1\theta$

T = torque in dyne-cm
k1 = torsion spring constant in dyne-cm per degree deflection
θ = angular deflection in degrees
Rb = radius of bob (cm)
L = length of bob (cm)

Shear Rate for Couette flows

$$\gamma = \frac{\pi \times rpm}{15\left[1-\left(\frac{Rb}{Rc}\right)^2\right]},\ sec^{-1}$$

where γ = shear rate in sec^{-1} at corresponding rpm
rpm is revolution per minute of cup
Rb is radius of bob (cm)
Rc is radius of cup (cm)

Rheology
Power Law Model

- Fracturing fluids have non-Newtonian flow behavior
- Mathematical model used to describe fluid viscosity in various environments that occur in the fracturing process
  - Power Law Model

FANN 50- Power Law Viscosity

For Power Law viscosity use the following formula:

$$\mu = \frac{k' \times 47880}{SR^{(1-n')}}$$

SR= Shear Rate

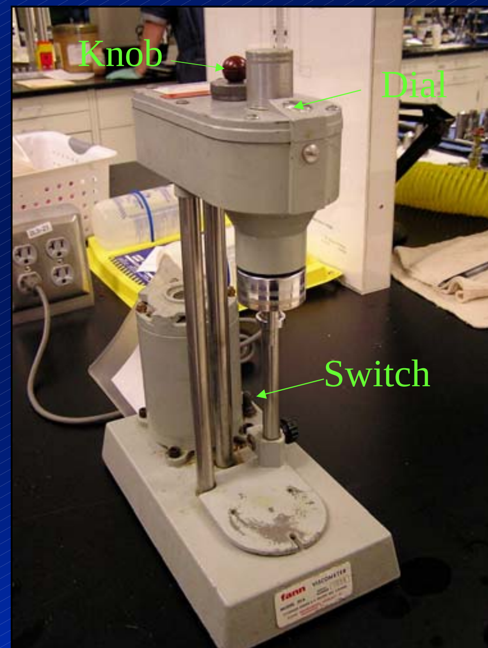
k'= Consistency index, interception with Log SS @ 1 s^{-1}

n'= Slope of the line on a Log (SR vs SS) Graph

FANN 35

Ability to test at 6 different speeds

Speed RPM	Viscometer Switch	Gear Knob
600	High	Down
300	Low	Down
200	High	Up
100	Low	Up
6	High	Center
3	Low	Center



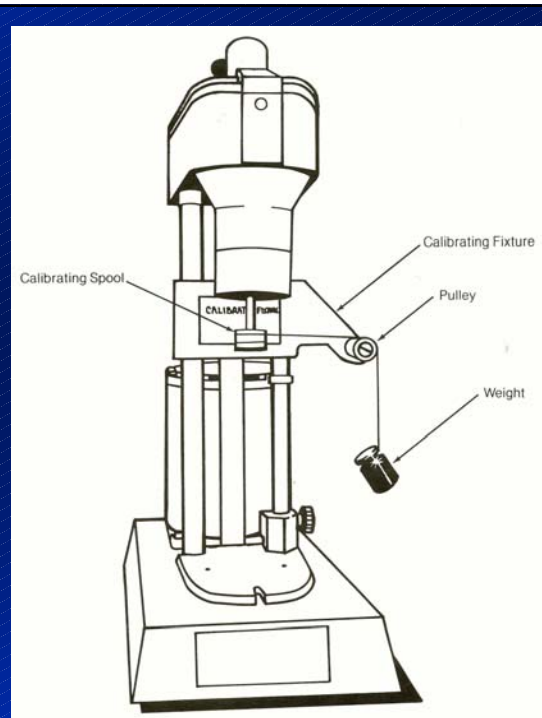
FANN 35 - Instrument Calibration

The calibration is checked by applying know torques to the bob shaft. For any applied torque, within the torque range of the spring, there should be a specific dial reading plus or minus a small tolerance.

Select the weight according to the following table, compare the dial reading with the one in the table. If necessary adjust the torsion spring

FANN 35 - Calibration

- *Calibrating spool*
- *Calibrating Fixture*
- *Pulley*
- *Weight*



FANN 35- Newtonian Viscosity

Newtonian viscosity in centipoise may be read directly from the dial when viscometer is run at 300 RPM with R1-B1-F1 combination. A Newtonian fluid should have the same viscosity at all shear rates

If other springs are used,then the dial reading has to be multiplied by the "f" factor
(spring constant from table 3)

Table 3

Deflection Ø With Various Calibration Weights for F/Torsion Spring Assemblies

Torsion Spring Assembly (with R1-B1 combination)	Torsion Spring Constant, K_1 Dynes/cm/deg def	Weight In Grams 10	20	50	100	200
		Dial Reading				
F-0.2	77.2	127.0	254.0	—	—	—
F-0.5	193.0	50.8	101.6	254.0	—	—
F-1	386.0	25.4	50.8	127.0	254.0	—
F-2	772.0	—	25.4	63.5	127.0	254.0
F-3	1158.0	—	—	43.0	84.7	169.4
F-4	1544.0	—	—	—	63.5	127.0
F-5	1930.0	—	—	—	50.8	101.6
F-10	3860.0	—	—	—	—	50.8

Factory tolerances for F1 spring only are 127 ± ½° for 50 g and 254 ± ½° for 100 g. A movement of ± ½° is permissible when the main shaft is turning. This movement will generally be dampened out when a fluid is being tested. Check the linearity of the dial reading with at least three weights. If the spring appears to be non-linear it is usually a sign that the bob shaft is bent. An instrument with these characteristics needs additional service and/or repair.

FANN 35- Newtonian Viscosity

For Newtonian viscosity use the following formula:

$\mu = S \times \Phi \times f \times C$

μ= Newtonian Viscosity - cP
S = Speed Factor (see table 5)
Φ= Dial reading
f= Spring Factor (see table 3)
C= Rotor -bob factor (see table 4)

Table 3

Deflection θ With Various Calibration Weights for F/Torsion Spring Assemblies

Torsion Spring Assembly (with R1-B1 combination)	Torsion Spring Constant, K_t Dynes/cm/deg def	Weight In Grams: 10	20	50	100	200
		Dial Reading				
F-0.2	77.2	127.0	254.0	—	—	—
F-0.5	193.0	50.8	101.6	254.0	—	—
F-1	386.0	25.4	50.8	127.0	254.0	—
F-2	772.0	—	25.4	63.5	127.0	254.0
F-3	1158.0	—	—	43.0	84.7	169.4
F-4	1544.0	—	—	—	63.5	127.0
F-5	1930.0	—	—	—	50.8	101.6
F-10	3860.0	—	—	—	—	50.8

Factory tolerances for F1 spring only are 127 ± ½° for 50 g and 254 ± ½° for 100 g. A movement of ± ½° is permissible when the main shaft is turning. This movement will generally be dampened out when a fluid is being tested. Check the linearity of the dial reading with at least three weights. If the spring appears to be non-linear it is usually a sign that the bob shaft is bent. An instrument with these characteristics needs additional service and/or repair.

TABLE 4

Rotor-Bob Comb.	R-B Factor C
R1 – B1	1.000
R1 – B2	8.915
R1 – B3	25.392
R1 – B4	50.787
R2 – B1	.315
R2 – B2	8.229
R2 – B3	24.707
R2 – B4	49.412
R3 – B1	4.517
R3 – B2	12.431
R3 – B3	28.909
R3 – B4	57.815

TABLE 5

Rotor rpm	Speed Factor S
.9	333.3
1.8	166.6
3	100
6	50
30	10
60	5
90	3.33
100	3
180	1.667
200	1.5
300	1.0
600	.5

Fann 35 Rotor / bob configuration

Factors used to calculate shear rate sec-1 from rpm

R1B1	1.72	Example - 100 rpm = 170 s^{-1}
R1B2	0.377	Example - 100 rpm = 37.7 s^{-1}

FANN 35- Newtonian Viscosity

For Newtonian viscosity use the following formula:

$$\mu = S \times \Phi \times f \times C$$

Example -

Rotor-Bob Configuration - R1B1
rpm - 300
Spring - 1
Dial Reading - 30

Calculate Viscosity - $\mu = 1 \times 30 \times 1 \times 1 =$? cP
Calculate Shear Rate - Shear rate = 1.72 x 300 = ? sec^{-1}

FANN 35- Newtonian Viscosity

For Newtonian viscosity use the following formula:

$\mu = S \times \Phi \times f \times C$

Example -

Rotor-Bob Configuration - R1B1
rpm - 300
Spring - 1
Dial Reading - 60

Calculate Viscosity - $\mu = 1 \times 60 \times 1 \times 1 = 60$ cP
Calculate Shear Rate - Shear rate = $1.72 \times 300 = 511$ sec^{-1}

FANN 35- Newtonian Viscosity

For Newtonian viscosity use the following formula:

$\mu = S \times \Phi \times f \times C$

Example -

Rotor-Bob Configuration - R1B2
rpm - 100
Spring - .2
Dial Reading - 11

Calculate Viscosity - $\mu = 3 \times 11 \times .2 \times 8.915 = ?$ cP
Calculate Shear Rate - Shear rate = $0.377 \times 100 = ?$ sec^{-1}

FANN 35- Newtonian Viscosity

For Newtonian viscosity use the following formula:

$\mu = S \times \Phi \times f \times C$

Example -

Rotor-Bob Configuration - R1B2
rpm - 100
Spring - .2
Dial Reading - 11

Calculate Viscosity - $\mu = 3 \times 11 \times .2 \times 8.915 = 59$ cP
Calculate Shear Rate - Shear rate = $0.377 \times 100 = 37.7\ \text{sec}^{-1}$

OFITE M900 Computerized Viscometer

FANN 50

The FANN 50 Rheometer, is a high precision, coaxial cylinder, rotational viscometer.

Can be used to characterize shear rate dependent rheological phenomena, like:

- ***Bingham Plastic flow - yield point then shear thinning***
- ***Pseudoplasticity (which include power law fluids) -shear thinning***
- ***Dilatancy - shear thickening***

Tests can be conducted under accurately controlled, shear rate, temperature and pressure

FANN 50

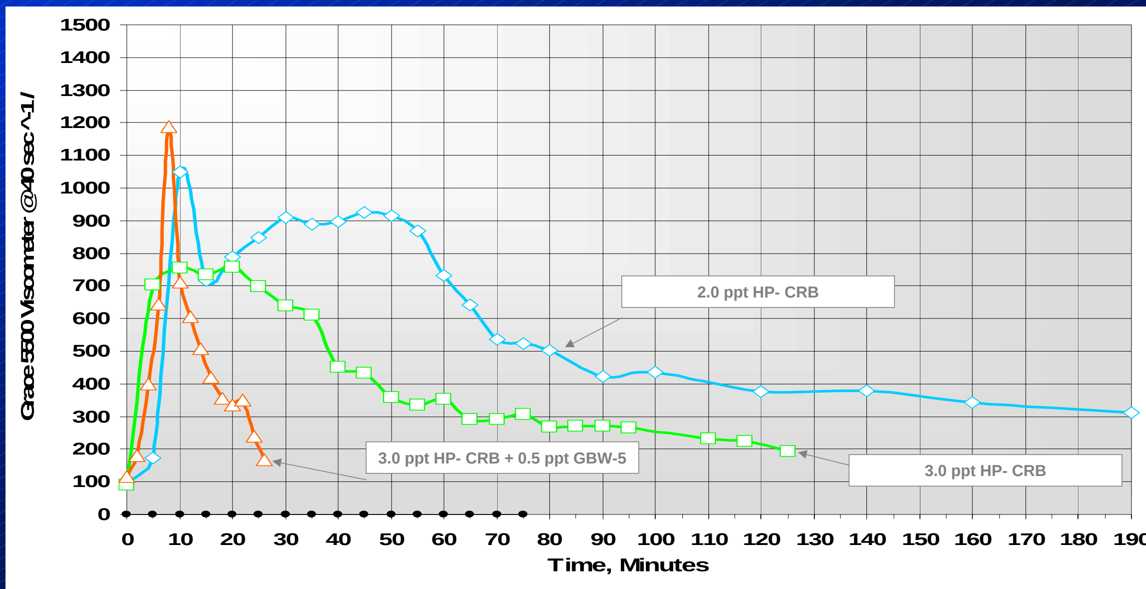
The FANN 50 Rheometer can measure

Shear stress Vs Shear Rate

Must be used in conjunction with the remote control option interface and a suitable Personal Computer.

The most common graph show during the test is the Viscosity Vs Temperature and time.


BJ Services
Data Monitor
Test In Progress
Set Time | Set # | Cycle # | Temperature | RPM | Torque | Rate | Stress | Viscosity
7.77 | 2 | 1 | 181.56 | 116.54 | 115034.30 | 99.09 | 820.78 | 828.29
SysA Viscosity Centipoise
System A Viscosity and Temperature vs. Time
SysA Temperature
Test In Progress
Set Time | Set # | Cycle # | Temperature | RPM | Torque | Rate | Stress | Viscosity
7.75 | 2 | 1 | 176.13 | 117.54 | 118574.07 | 99.95 | 846.75 | 847.21
SysB Viscosity Centipoise
System B Viscosity and Temperature vs. Time
SysB Temperature
Return



Fann 50

Rotational Concentric Cylinder

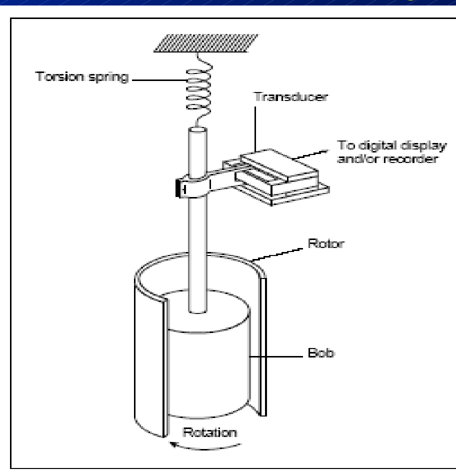


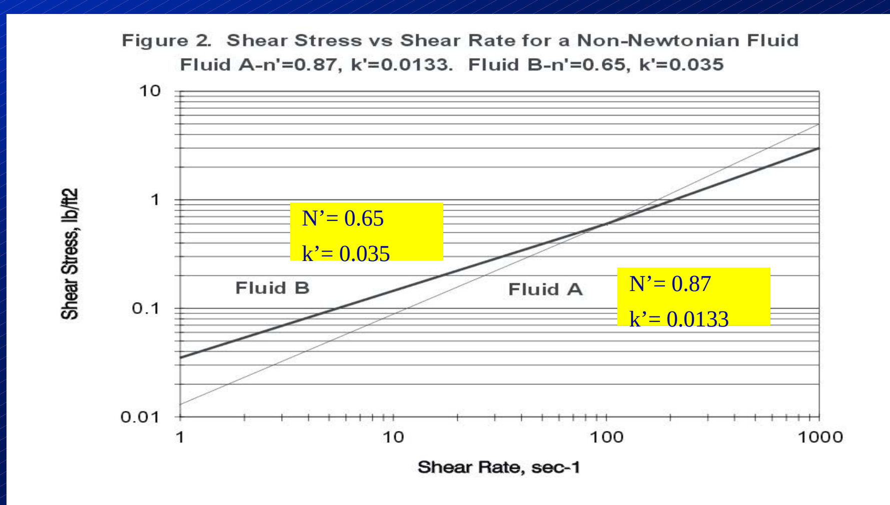
Figure 8-5. Rotational concentric cylinder viscometer.

Fann 50 Rotor / bob configuration

Factors used to calculate shear rate sec-1

R1B1	1.72
R1B5/B5X	0.85
R1B2	0.377

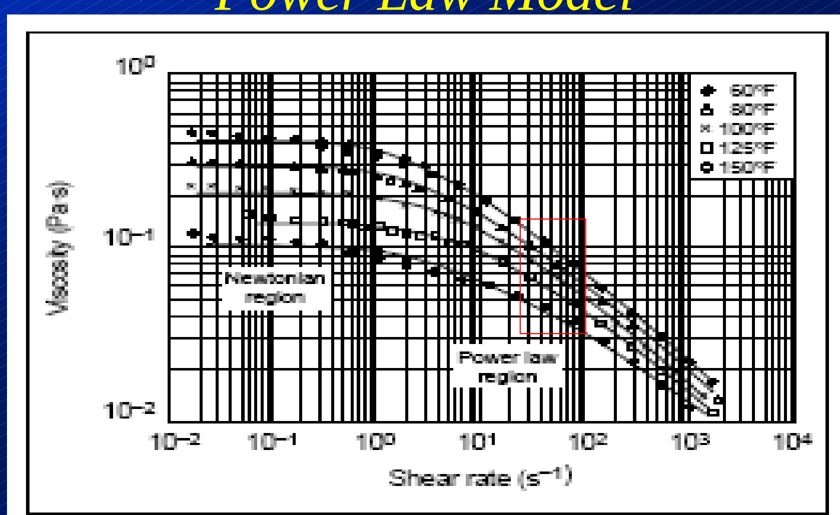
Rheology



Rheology
Power Law Model

- Fracturing fluids have non-Newtonian flow behavior
- Mathematical model used to describe fluid viscosity in various environments that occur in the fracturing process
  - Power Law Model

Rheology
Power Law Model



Rheology

Power Law Model

- Power Law Model $\tau = K\gamma^n$

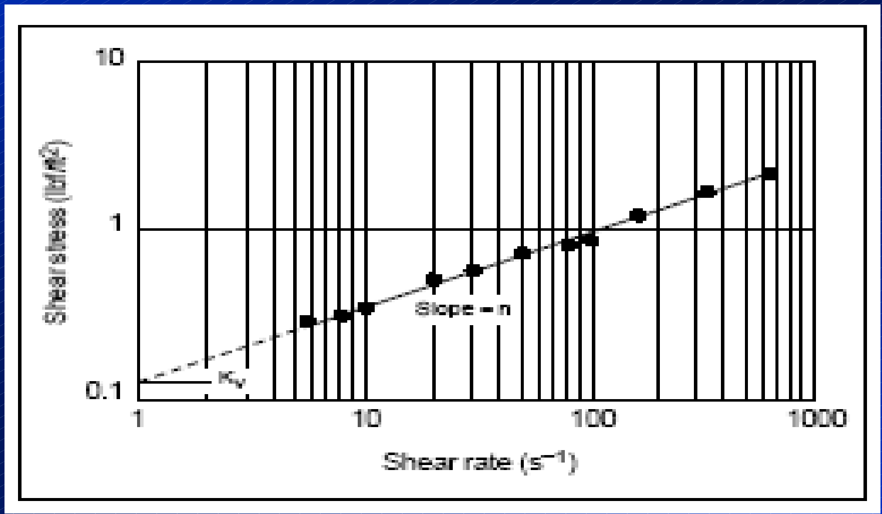
  Apparent Viscosity $\mu = \frac{\tau}{\gamma}$

  Apparent Viscosity $\mu = \frac{K}{\gamma^{(1-n)}}$

- Plot log μ (ap. viscosity) vs log γ (shear rate)

Rheology

Power Law Model



Rheology

Power Law Model

- Power Law Model $\tau = K\gamma^n$
  - K - consistency index (lbf-s^n/ft^2)
  - n - flow behavior index (dimensionless)

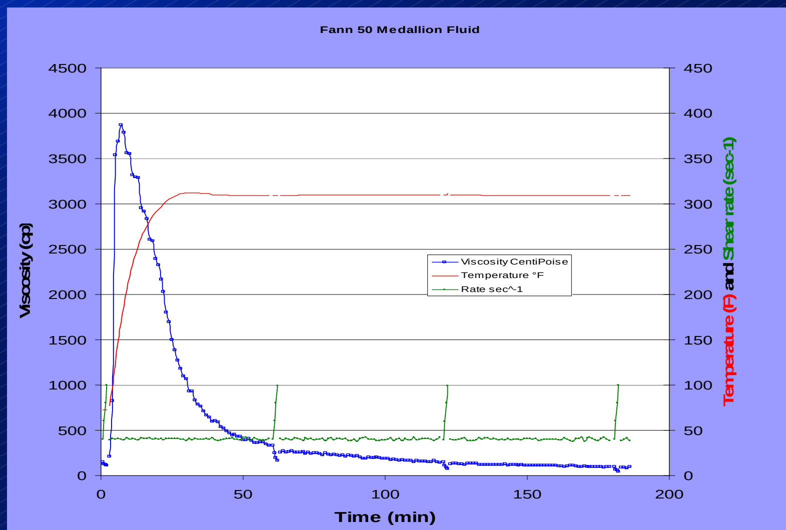
- Plot log τ (shear stress) vs log γ (shear rate)
  - K - value of τ at 1 sec^{-1}
  - n - slope

Rheology

Power Law Model

- n decreases
  - velocity profile flattens, fluid moves with fixed velocity, particles segregation reduced
  - fluid becomes more shear thinning causing reduced friction pressure
- K increases
  - larger K the more viscous the fluid for specific n
  - viscosity increases retarding particle settling

Fann 50 Rheology



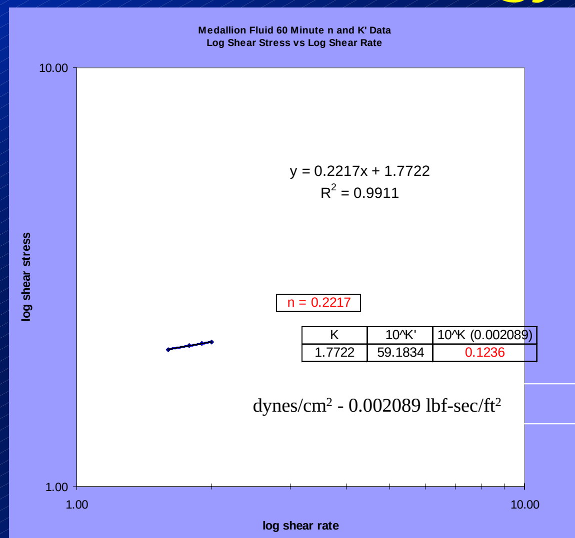
Fann 50 Rheology n and K

Time	Temperatur	Stress	Rate	Viscosity	n	K	corr	Kv	Kp	Kf
Min	°F	dyne/cm^2	sec^-1	CentiPoise		lbf*s^n/ft^2		lbf*s^n/ft^2	lbf*s^n/ft^2	lbf*s^n/ft^2
60.6	309	133.23	40	333	**0.2217**	0.11228	0.9955	**0.12359**	0.12911	0.13332
61.1	309	148.65	60	247						
61.6	309	155.93	80	195						
62.1	309	163.51	100	165						

Shear Rate	Stress
sec^-1	dyne/cm^2
40.0	133.23
60.3	148.65
80.1	155.93
99.4	163.51

log Shear Rate	log Shear Stress
sec^-1	dyne/cm^2
1.60	2.12
1.78	2.17
1.90	2.19
2.00	2.21

Fann 50 Rheology



Fann 50 Rheology
n and K

Time	Temperature	Stress	Rate	Viscosity	n	K	corr	Kv	Kp	Kf
Min	°F	dyne/cm^2	sec^-1	CentiPoise		lbf*s^n/ft^2		lbf*s^n/ft^2	lbf*s^n/ft^2	lbf*s^n/ft^2
60.6	309	133.23	40	333	**0.2217**	0.11228	0.9955	**0.12359**	0.12911	0.13332
61.1	309	148.65	60	247						
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62.1	309	163.51	100	165						

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sec^-1	dyne/cm^2
40.0	133.23
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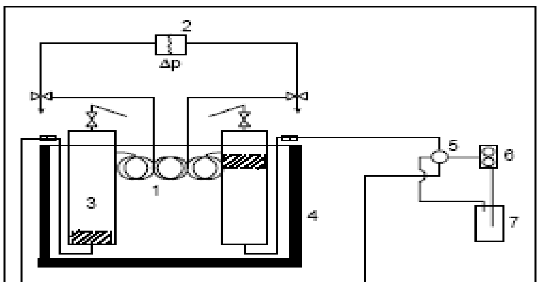
99.4	163.51

log Shear Rate	log Shear Stress
sec^-1	dyne/cm^2
1.60	2.12
1.78	2.17
1.90	2.19
2.00	2.21

Fann 50 Rheology n and K

Time	Temperature	n	K	Kv	corr	Viscosity 40 sec-1	Viscosity 100 sec-1	Viscosity 170 sec-1
Min	°F		lbf*s^n/ft^2	lbf*s^n/ft^2		CentiPoise	CentiPoise	CentiPoise
2.1	73	0.639	0.0116	0.01217	1	147	105	87
62.1	309	0.2217	0.11228	0.12359	0.9955	304	149	99
122.1	311	0.2264	0.05085	0.05595	0.9916	140	69	46
182.1	309	0.2186	0.03312	0.03646	0.9973	89	43	29

RCV



1 Measurement capillary
2 Differential pressure Δp transducer
3 Floating piston accumulator containing test fluid on top of piston and hydraulic fluid below piston
4 Programmable-temperature oil bath
5 Hydraulic fluid directional control valve
6 Variable rate positive-displacement pump
7 Hydraulic fluid reservoir

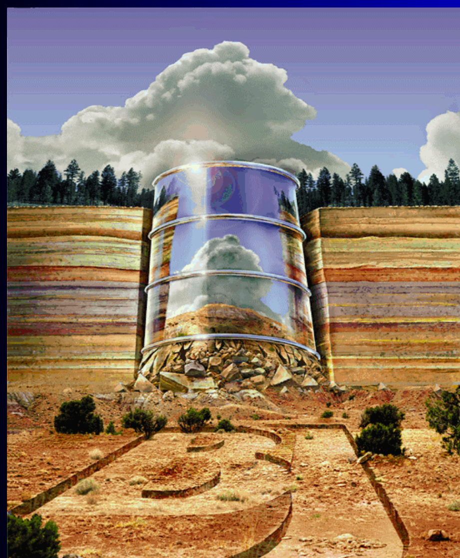
Figure 8-6. Reciprocating capillary viscometer.

Grace Instruments Model 5500

This is a benchtop rheometer and has the same geometry as FANN 50

Friction Loop Testing (Ambient Temperature)

Foam Loop Testing (Up to 275 °F)



Cementing Overview

Presented by

BJ Services Company

Updated: Nov. 2006

Discussion Topics

- API Testing
- Laboratory Testing
- Lunch
- Laboratory Tour
- Test

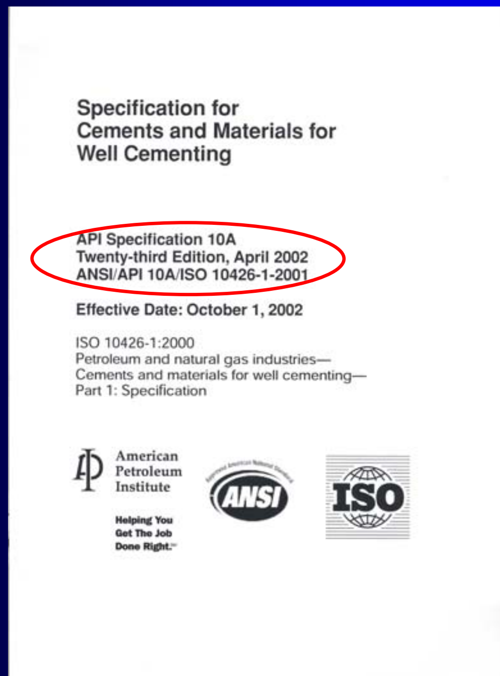
Cement Properties

- Most API Tests Focused on Slurry Properties Prior to Set
  - Thickening time,
  - Fluid loss,
  - Rheology,
  - Free water and
  - Gas migration, (not API test)
- Only Set Cement Test was the Unconfined Compressive Strength (UCS) Test
  - More was better
  - “Rules of Thumb” applied for minimum values

API Testing

API Specifications and Recommendations

- API Spec 10A (October 2002)
  - ISO 10426-1
- API RP 10B (2004)
  - ISO 10426-2
- ISO 10426-3
  - Testing Deepwater well Cement
- ISO 10426-4
  - Preparation and Testing of Atmospheric Foamed Cement



Specification for
Cements and Materials for
Well Cementing

API Specification 10A
Twenty-third Edition, April 2002
ANSI/API 10A/ISO 10426-1-2001

Effective Date: October 1, 2002

ISO 10426-1:2000
Petroleum and natural gas industries—
Cements and materials for well cementing—
Part 1: Specification

American
Petroleum
Institute

Helping You
Get The Job
Done Right.

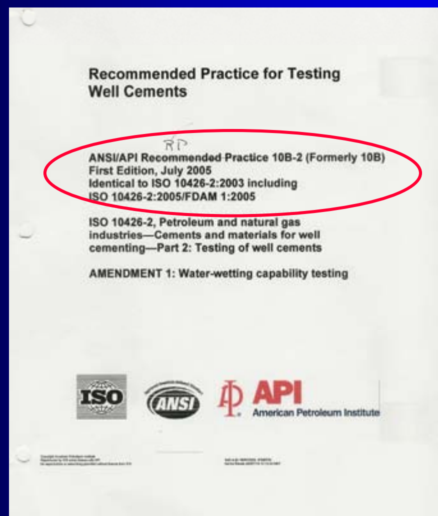
ANSI

ISO

API Specifications for Cements and Materials for Well Cementing

- API Spec 10A (October 2002)
  - ISO 10426-1:2001
  - Specifies Chemical, Physical and Performance requirements for API cements and neat slurries.
    - MgO, SO3, C3A, C3S, C4AF
    - Loss on ignition, Insoluble residue, Fineness
    - Compressive Strength, Free Water, Thickening Time
  - Specifies required equipment
    - Calibration procedures
    - Testing procedures

API Testing



Recommended Practice for Testing
Well Cements

ANSI/API Recommended Practice 10B-2 (Formerly 10B)
First Edition, July 2005
Identical to ISO 10426-2:2003 including
ISO 10426-2:2005/FDAM 1:2005

ISO 10426-2, Petroleum and natural gas
industries—Cements and materials for well
cementing—Part 2: Testing of well cements

AMENDMENT 1: Water-wetting capability testing

ISO ANSI API American Petroleum Institute

API Recommended Practice for Testing Well Cements

- API RP 10B-2 (December 2005)
  - ISO 10426-2
  - Specifies procedures for testing and determining properties of cement slurries and set cement.
    - Sampling, Preparation, Density
    - Compressive Strength, Thickening Time, Fluid Loss
    - Permeability, Rheology, Stability, Compatibility
  - Specifies required equipment
    - Calibration procedures
    - Testing procedures

BJ Laboratories

- Distric Laboratories
- Region Laboratories
- Tomball Laboratories

District Laboratories

- Testing Capabilities
  - Thickening Time
  - Rheologies (Ambient and Temperature)
  - Free Water
  - Fluid Loss (< 190F)
  - Destructive Compressive Strength

District Laboratories

- Testing Equipment
  - Atmospheric Consistometer
  - HPHT Consistometer
  - Fann 35 (viscometer)
  - Conventional Fluid Loss Cell (5 - 10 inch)
  - Curing Chamber / Hydraulic press

Slurry Preparation and Conditioning

- Mixing
  - Mix the water, cement and additives in API mixer (Waring blender) at low speed
    - 4,000 rpm during 15 seconds
  - Shear at high rate
    - 12,000 rpm for 35 seconds

Slurry Preparation and Conditioning (cont.)

- **Conditioning**
  - Simulates slurry agitation
  - Place slurry in consistometer and continue stirring while heating up to BHCT and pressuring up to BHP

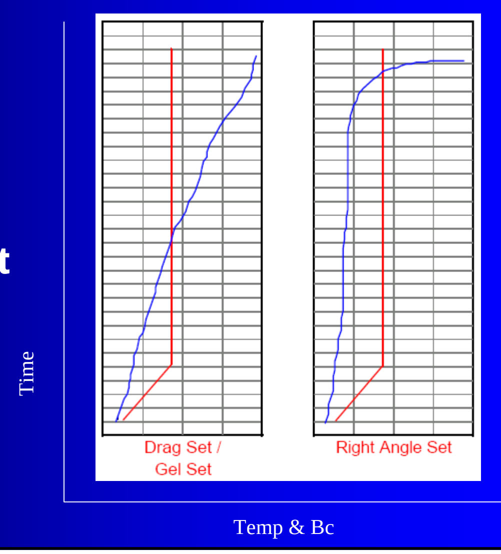
API Mixer

Thickening Time

- Thickening / Pump Time
  - Measured by Consistometer
    - Atmospheric (BHCT < 194 °F)
    - Pressurized
  - Bearden units of consistency, B_c
    - Related to torque imparted on the paddle shaft
    - Measured with a voltage potentiometer
    - Slurry is usually considered unpumpable at 70 – 100 B_c
  - Test is performed at BHCT
    - Conditioning time, heat-up rate and pressure are determined by API Spec 10 tables
    - Once BHCT is reached, it is maintained constant

Thickening Time (cont.)

- Types of slurry:
  - Gel Set
    - Drag Set
  - Right Angle Set



Thickening Time (cont.)

- Batch Mixing
  - Slurry is conditioned (stirred) at atmospheric conditions to simulation batch mixing time
    - Typically one hour
  - Reported thickening time does not include batch mix time
- Hesitation Squeeze
  - Second temperature heat-up (ramp) from BHSqT to BHST
  - Slurry stirring is cycled on/off during second temperature ramp to simulate hesitation method
  - Generally gives shorter thickening time than Continuous Pumping Squeeze

Atmospheric Consistometer

Consist of a stainless steel water bath that houses a slurry container

Temperatures from ambient to 194 F

Rotation of the slurry containers at 150 RPM

Atmospheric Consistometer

Pressurized Consistometer

Consist of a rotating cylindrical slurry container with a stationary paddle assembly, in a pressure chamber.

Working pressure & Temp is determined by the drive assembly
- Packing Drive: 25K psi @ 400 F
- Magnetic Drive: 40K Psi @ 600 F

Pressurized Consistometer

Pressurized Consistometer Parts

Rheology

- Rheology is the study of flow and deformation of fluids
- Needed to calculate friction pressures and to predict flow regimes
- Rheology is the relationship between flow rate (shear rate) and the pressure (shear stress) needed to move a given fluid
  - Shear Rate (SR) = difference in velocity of two fluid particles divided by the distance between them
  - Shear Stress (SS) = frictional force created by the two fluid particles rubbing against each other

Fann-35 Rotational Viscometer

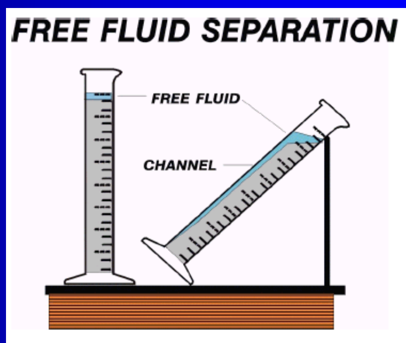
- Stationary cup and rotating sleeve
- Internal shaft & bob
- Shear Rate
  - Proportional to rotational speed
  - Shear Rate = 1.7023 X RPM
- Shear Stress
  - Proportional to torque imparted on shaft
  - Shear Stress = 1.065 x Dial Reading

Fann-35 Rotational Viscometer (cont.)

- Measurements are generally taken at ambient temperature (to simulate mixing conditions) and BHCT (to simulate pumping conditions)
- General Rules of Thumb
  - Low end readings (3 & 6 rpm) of less than 5 indicate the possibility of solids settling
  - A low end reading of greater than 40 indicates a strong possibility of gelation
  - High end readings (300 & 600 rpm) of greater than 300 could indicate difficulty in field mixing and pumping

Free Fluid Test

- Mix and condition slurry to BHCT
- Pour slurry into 250 mL graduated cylinder
- Leave static 2 hours at ambient temperature
- Either 90° or 45°
- Measure free fluid with a pipette



Fluid Loss

- Fluid loss is the rate at which water will be forced out of the cement slurry into permeable formations, expressed in mL/30 min
- Measured with a Fluid Loss Cell
  - Slurry is mixed and conditioned to BHCT
  - Cell consists of a pressurized cylinder with a 325 mesh screen insert to simulate permeable formations
  - 1,000 psi pressure differential is applied
  - Filtrate is collected during a 30 minute interval and measured
  - Standard Cell or Stirred Fluid Loss Cell

Standard Fluid Loss Test

- BHCT < 194 °F
- Method 1 (Standard Fluid Loss Cell)
  - Condition slurry to BHCT in pressurized consistometer
  - Heat slurry to Temp
  - Transfer slurry to pre-heated fluid loss cell
  - Increase temperature to BHCT
  - Perform fluid loss test

Standard Fluid Loss Cells & Parts

Compressive Strength

The strength of the cement is the resistance it offers to being crush (compressive strength) or pulled apart (tensile Strength)

The compressive strength are reported in PSI

Compressive Strength

Most authorities agree the following:

- **5 to 200 psi is adequate to support casing**
- **500 psi is adequate for drill-out**
- **1000 psi to perforate**
- **2000 psi for stimulation**
- **To side track - More than adjacent formation**

Destructive Compressive Strength

- **Pressure Curing Chamber**
- **The hydraulic press**

Pressure Curing Chamber

- Stainless Steel Cylinder that Threads into the chamber
- Capacity is 8 cubes
- Max Working pressure is 3000 psi at 500F
    - Prepare conditioned slurry in 2 in^2 cube molds
    - Cure in curing chamber to BHS

Pressure Curing Chamber

Hydraulic Press

•A load is applied to compress the sample

•Maximum load is recorded when the sample crush

Load to failure in hydraulic press at different elapsed times

•The compressive strength is is reported in psi

UCS = Force / Area

Hydraulic Press

Hydraulic Press

Hydraulic Press

Region Laboratories

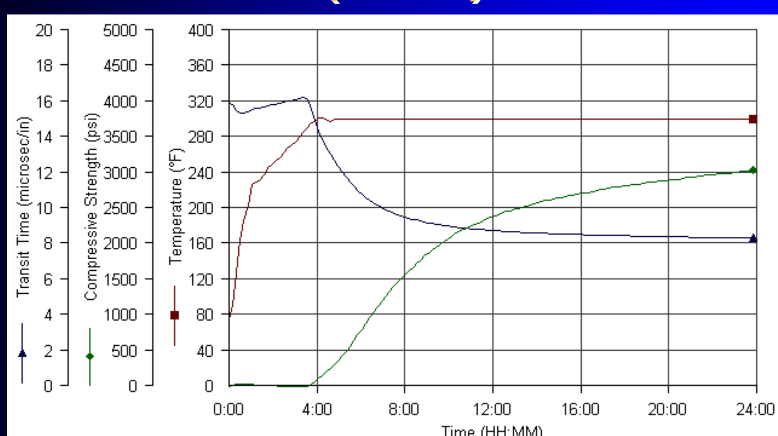
- Testing Equipment
  - HPHT Consistometer
  - Atmospheric Consistometer
  - Fann 35 (viscometer)
  - Conventional Fluid Loss Cell (5 - 10 inch)
  - Curing Chamber
    - Ultrasonic Cement Analyzer (UCA)
    - BP Settling Tube
    - Stirred Fluid Loss Cell
    - Paddle, Mixer and LS Additives
    - Gas Migration Apparatus
    - Tensile and flexural strength Machine (Gilson)

Ultrasonic Cement Analyzer

- Non-destructive test
- Measures and records the inverse P-wave of velocity through a cement slurry as a function of time
- Unconfined compressive strength is estimated via an empirical algorithm
- Continuous read-out
- Also plots sonic travel time, in order to calculate attenuation time to calibrate cement bond logs

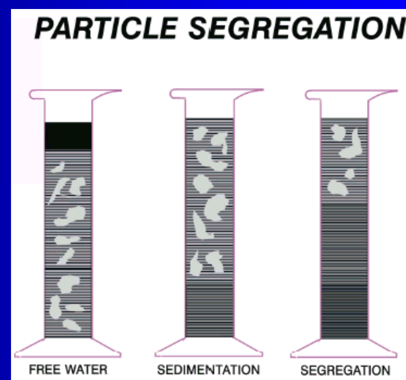
Ultrasonic Cement Analyzer (cont.)

Ultrasonic Cement Analyzer (cont.)



Slurry Segregation & Settling

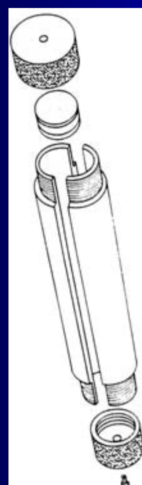
- Test measures the ability of the slurry to maintain a stable suspension at downhole conditions
- Critical for deviated and horizontal wellbores and for gas-migration control slurries



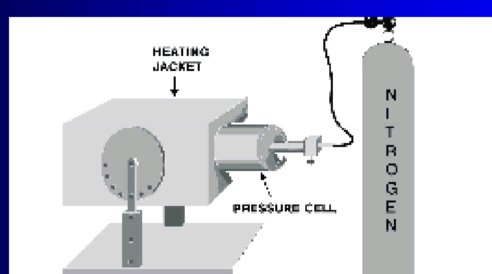
BP Settling Test

- Mix and condition slurry to BHCT or 194 °F
- Transfer to preheated settlement tube
- Place in pre-heated curing chamber, ramp to BHST (if needed) and maintain for 24hrs, applying pressure
- After test period, cool to ambient temperature
- Measure settlement, in mm
- Break column into 3/4” to 1” segments
- Measure density of each segment by Archimedes method (dipping in water)

BP Settling Test



Heating Apparatus for setting tube



2000 psi / 500 F

Fluid Loss Test at High Temperature

This apparatus is designed to condition the slurry under dynamic condition and determine the fluid loss under static condition without having to handle the slurry

Fluid Loss Test at High Temperature

- BHCT > 194 °F
- Method 2 (Stirred Fluid Loss Cell)
  - Condition slurry to BHCT in stirred fluid loss cell
  - Invert cell
  - Apply differential pressure
  - Perform fluid loss test
- Safer, Easier & More Representative

Stirred Fluid Loss Cell

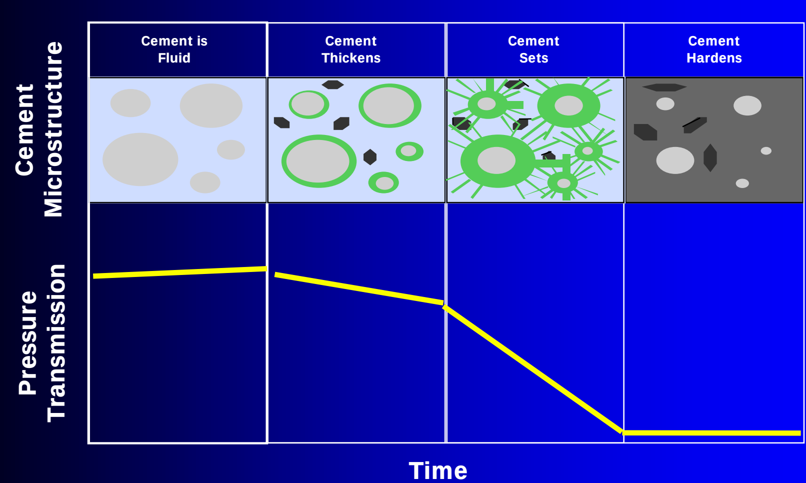
GAS MIGRATION

GAS MIGRATION
Two Types

- Primary
  - Occurs minutes - hours after completion of cementing operation
  - Related to the cementing operation itself

- Secondary
  - Occurs weeks, months or years after the well was cemented
  - Should be considered as leakage

GAS MIGRATION
Cement Hydration and Pressure Transmission



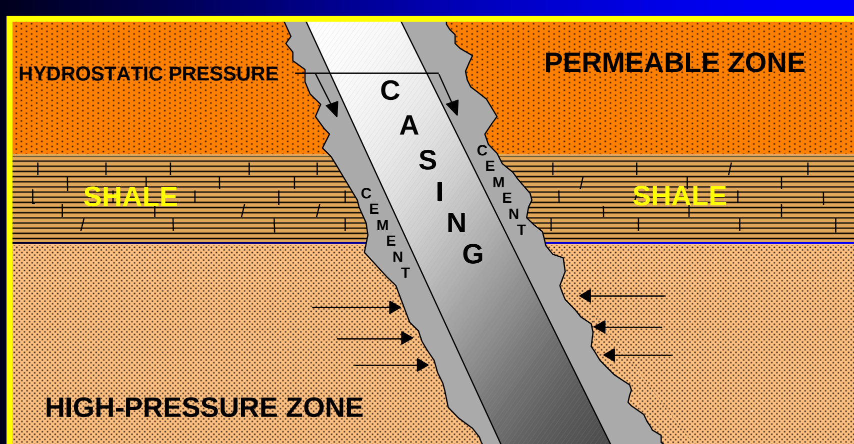
GAS MIGRATION

Causes

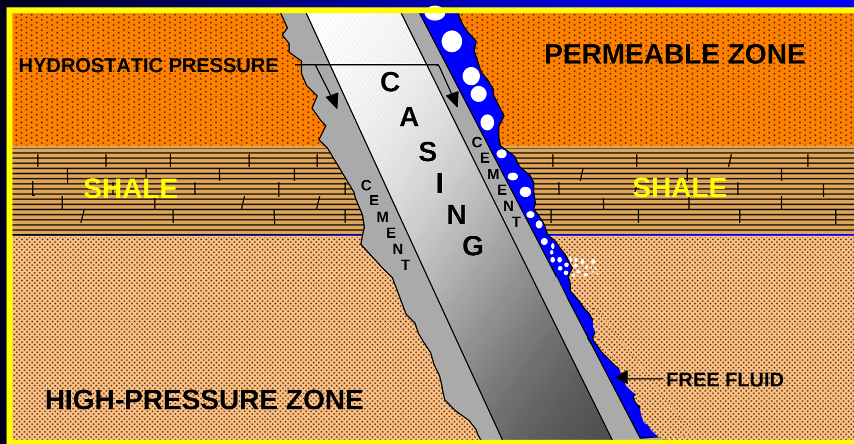
- **Poor Mud Removal**
- **Bridging**
- **Excess Free Fluid**
- **Particle Segregation**
- **Lack of Fluid Loss Control**
- **Lack of Permeability Reduction**

Could be prevented by the use of proper mechanisms to control Gas

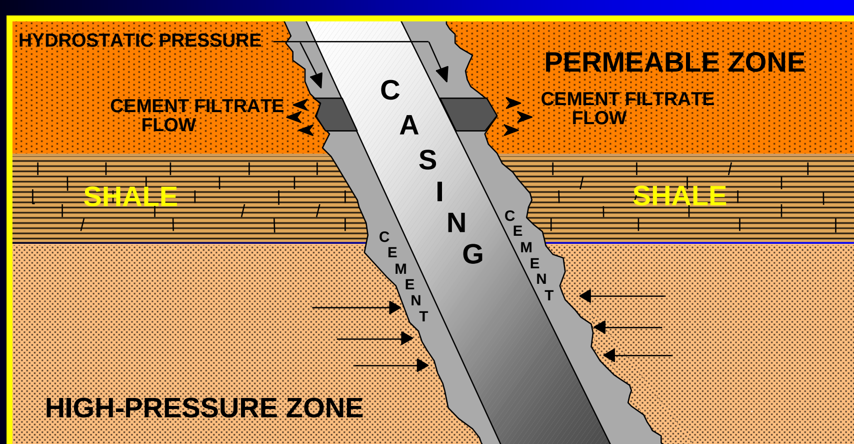
GAS MIGRATION

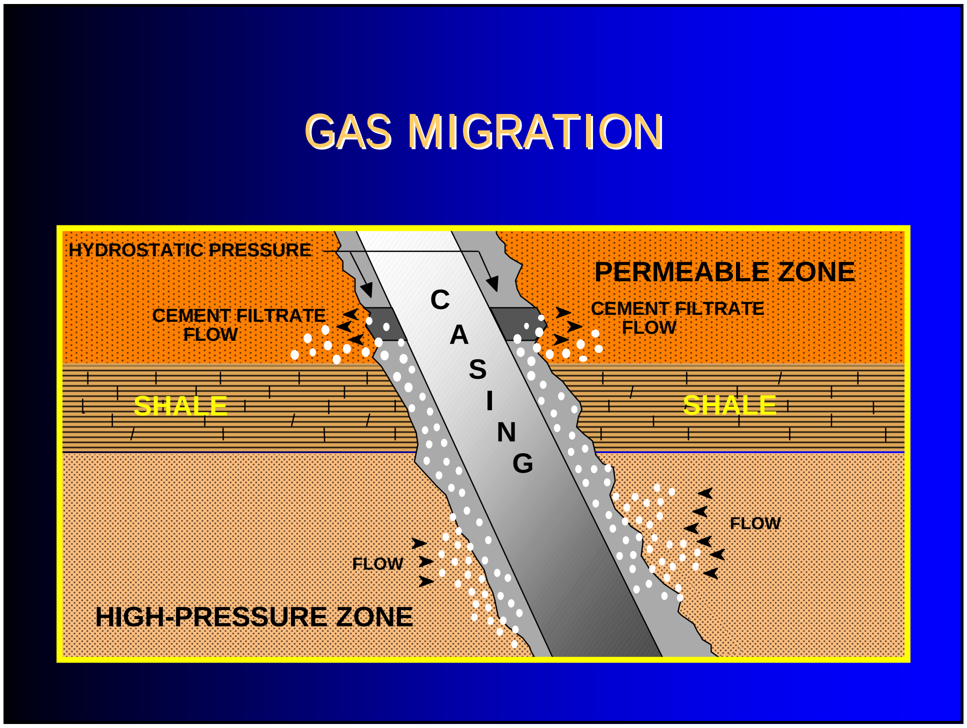


GAS MIGRATION



GAS MIGRATION

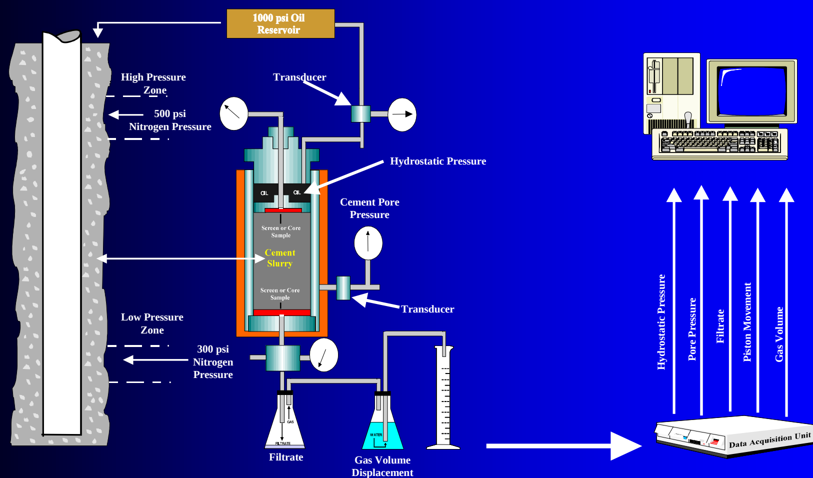



GAS MIGRATION
HYDROSTATIC PRESSURE
PERMEABLE ZONE
CEMENT FILTRATE
FLOW
CEMENT FILTRATE
FLOW
CASING
SHALE
SHALE
FLOW
FLOW
HIGH-PRESSURE ZONE


GAS MIGRATION
BJ Services' Automated Gas Flow Model
High Pressure Zone
Pore Pressure
LVDT (Piston Travel)
Low Pressure Zone
(Back Pressure Regulator)
Heating
Jacket

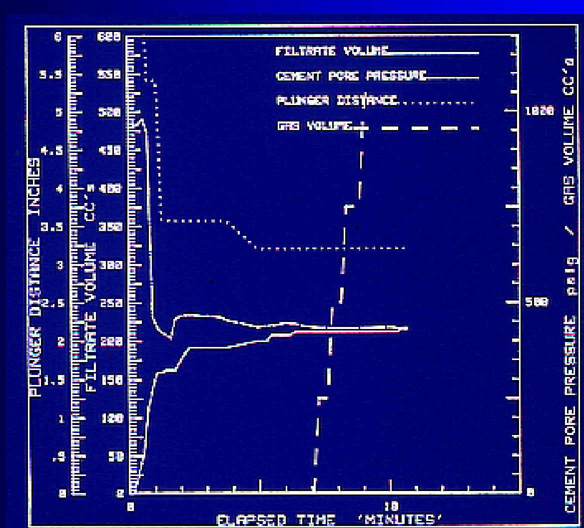
GAS MIGRATION

GAS FLOW MODEL



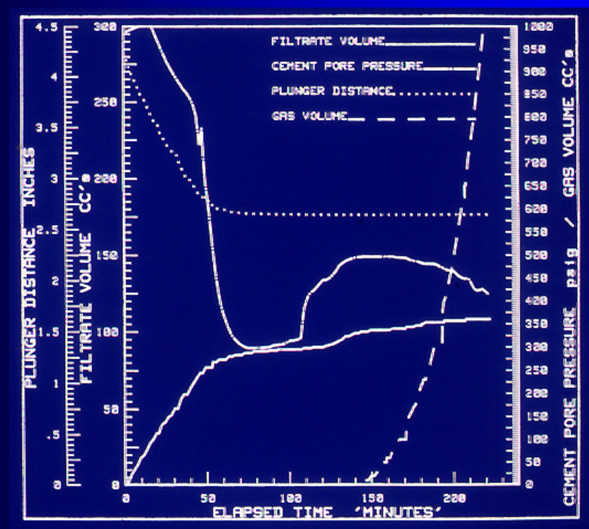
Class H + Retarder @ 16.5 ppg

T.T. = 3:05; F.L. = 1200 cc's; F.W. = 1.5 cc's; BHST = 180°F



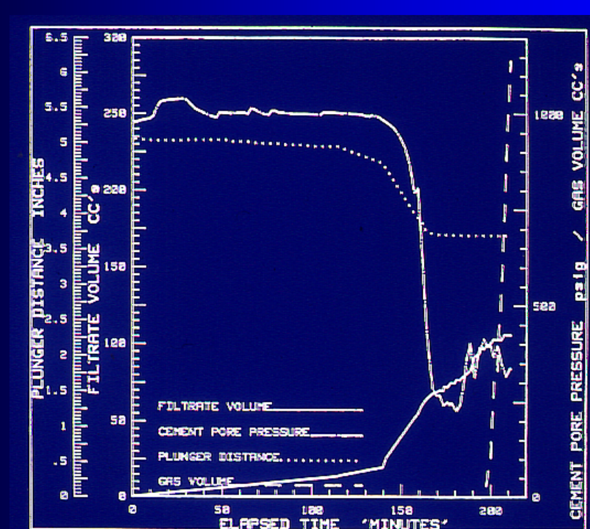
Class H + 1.2% FLA + Retarder @ 16.5 ppg

T.T. = 3:28; F.L. = 126 cc's; F.W. = Trace; BHST 180°F



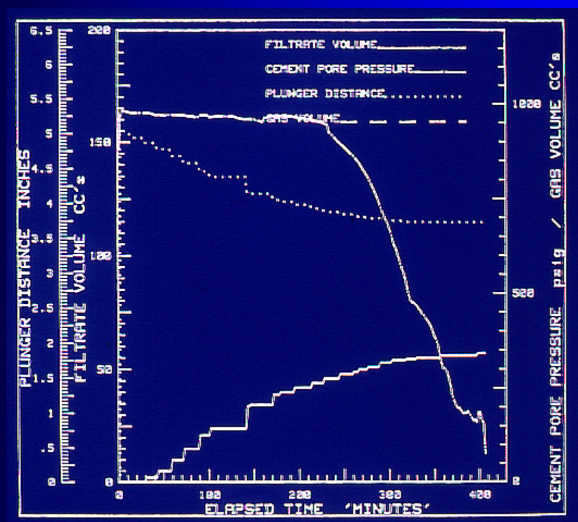
Class H + 2.0% FLA + Retarder @ 16.5 ppg

T.T. = 3:45; F.L. = 12 cc's; F.W. = Zero; BHST 180°F



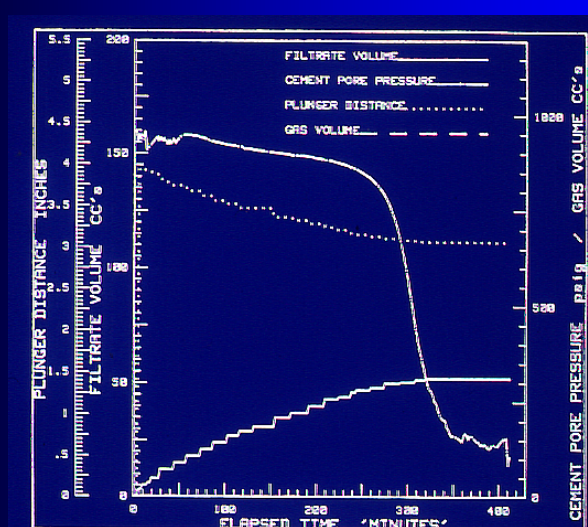
Class H + 1.5% GCA + Retarder @ 16.2 ppg

T.T. = 3:31; F.L. = 35 cc's; F.W. = Zero; BHST = 180°F

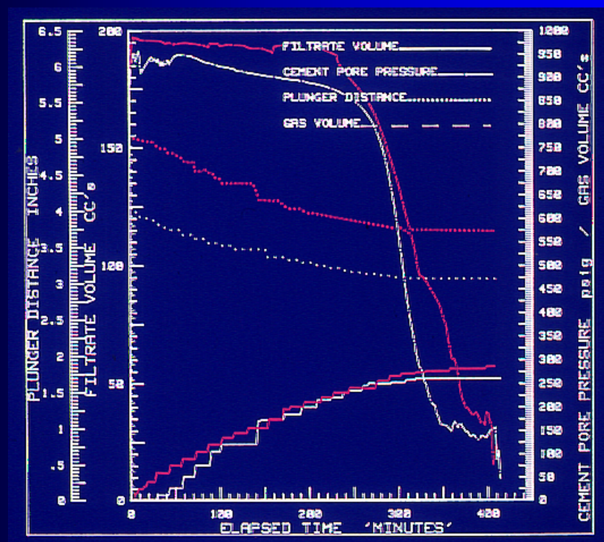


Class H + 1.5% GCA + Retarder @ 16.2 ppg

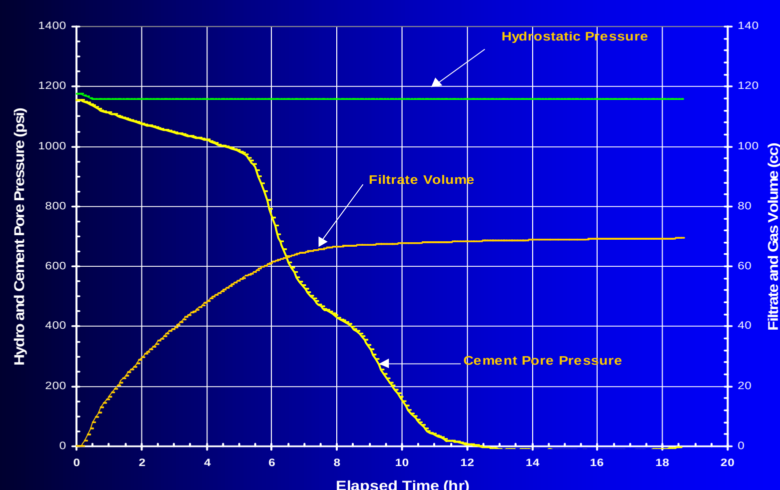
T.T. = 3:47; F.L. = 41 cc's; F.W. = Trace; BHST = 180°F



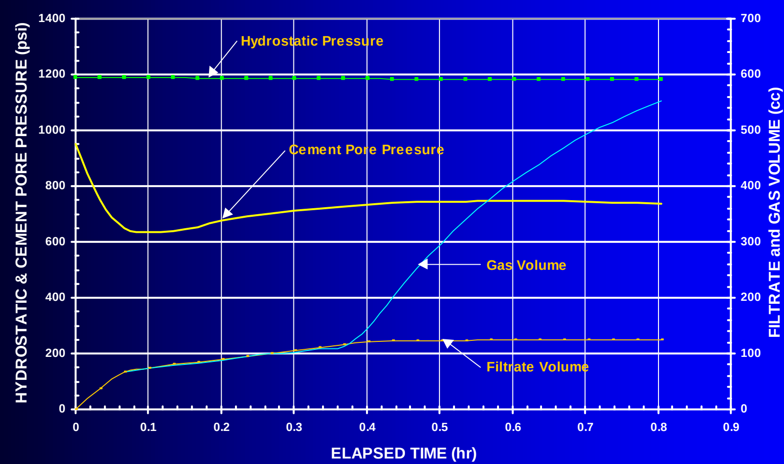
Repeated Test, Chart Overlapped for Consistency



GAS MIGRATION



GAS MIGRATION



Gas Migration
Prevention

- Zero Free Fluid
- Fluid Loss < 50 Cc's
- Short Transition Time (Right Angle Set)
  - Not Necessary for Gas Control
  - Necessary to Control Water Flow
- Minimize Shrinkage
- Use Appropriate Mechanism

Gas Migration Mechanisms

BJ provides a complete line of additives to covers all mechanisms for gas migration control at temp. from 40° to 450°F

- Film forming
  - BA-86L
  - BA-11
  - BA-10
- Bridging
  - BA-58 & BA-58L
  - BA-90
  - BA-100 & BA-100L
- Expansive
  - EC-1 / EC-2
  - BA-61 / BA-59
  - Foam Cement
- Short Transition / Thixotropic
  - DeepSet™
  - Custom designs for specific well conditions

Tensile and Flexural strength Machine

Tensile Strength Fixture

Flexural Strength Fixture

TTC Laboratory

- Testing Capabilities
  - Thickening Time
  - Rheologies (Ambient and Temperature)
  - Free Water (< 190F)
  - Fluid Loss (< 190F)
  - Destructive Compressive Strength
  - Non-Destructive Compressive Strength
  - Free Water (> 190F)
  - Fluid Loss (> 190F)
  - Liquid Stone
  - Gas Migration

•Gel Strength Determination
•Cement Expansion/Shrinkage
•Mechanical Properties of Cement
•Particle Size Analysis
•Spacer / Mud Wettability
•Loss Circulation
•Heat of Hydration

Multiple Analysis Cement Analyzer (MACS)

Measures the gel strength of a slurry from 100 to 500 Lb/ 100 sqft

Maximum Temperature : 500 F

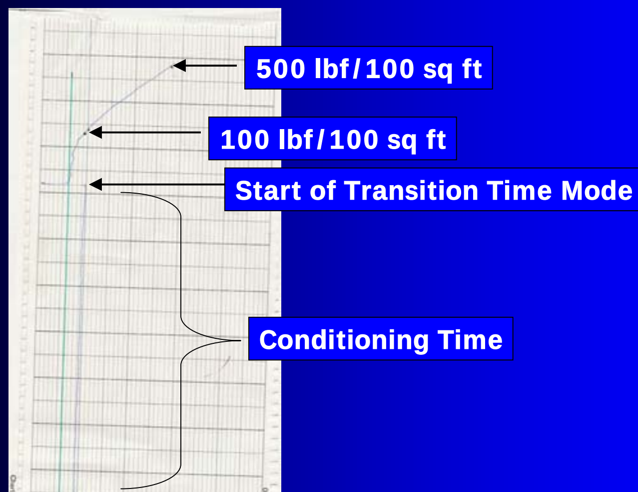
Maximum pressure : 20,000 psi

Measure how rapid the cement can go from a liquid state to a condition where it does not transmit hydrostatic pressure

Multiple Analysis Cement Analyzer (MACS)

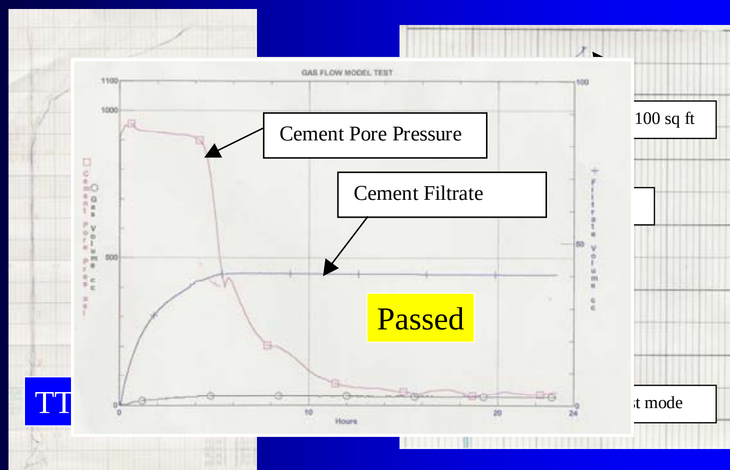
Table Top SGS Tester

Transition Time Chart

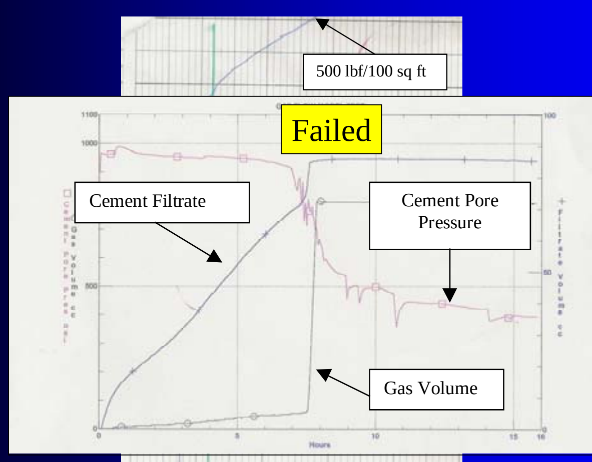


Transition Time vs Gas Migration

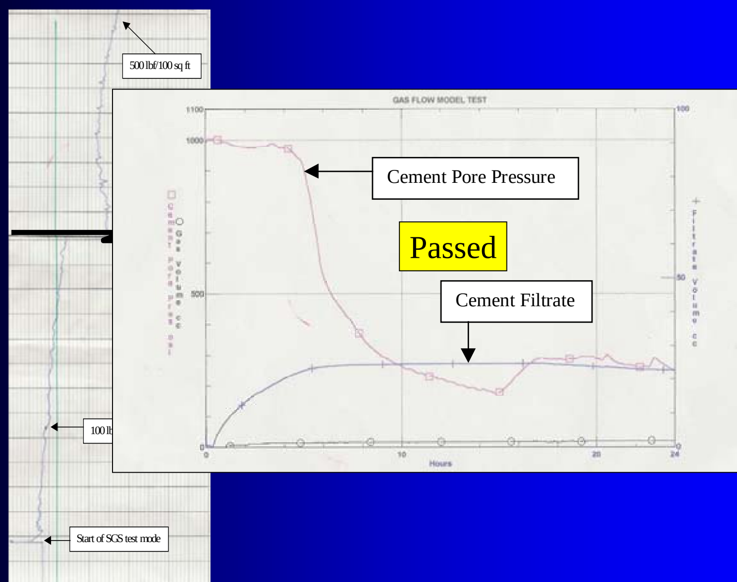
Slurry 1 Evaluation



Slurry 2 Evaluation



Slurry 3 Evaluation



BJ Expansion Apparatus

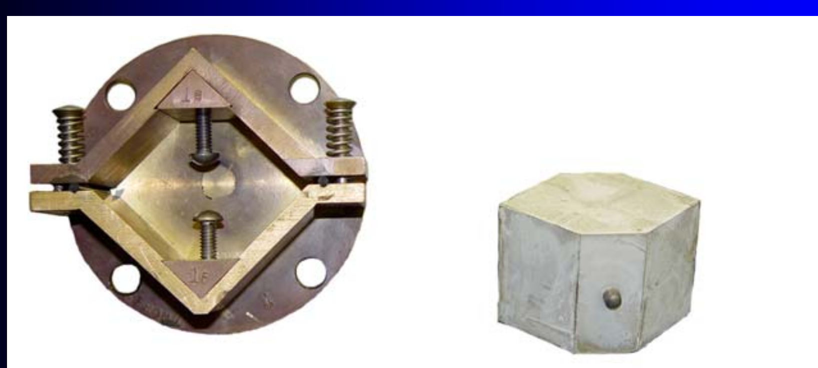
Measures the expansion or contration of a slurry under pressure and temperature conditions

Maximum Temperature : 500 F

Maximum pressure : 4,000 psi

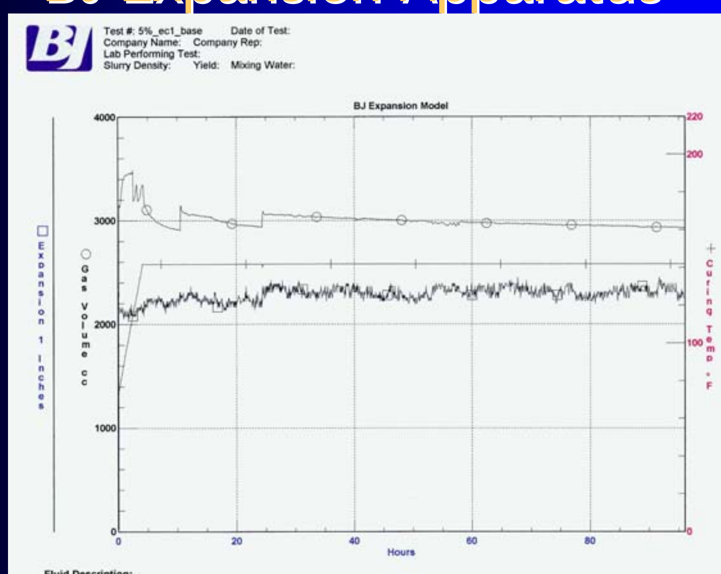
BJ Expansion Apparatus

Cement Expansion / Shrinkage (cont.)



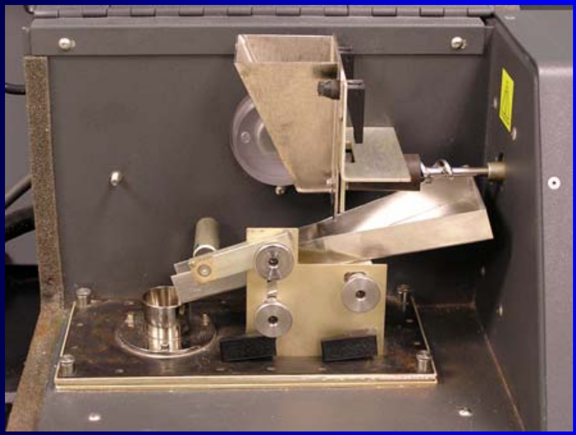
BJ Expansion Apparatus

BJ Expansion Apparatus

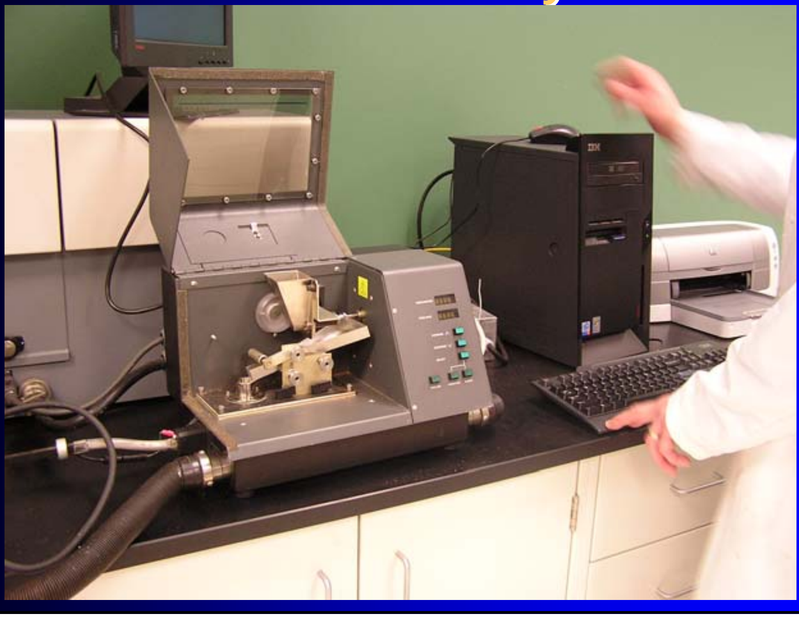


Particle size analyzer

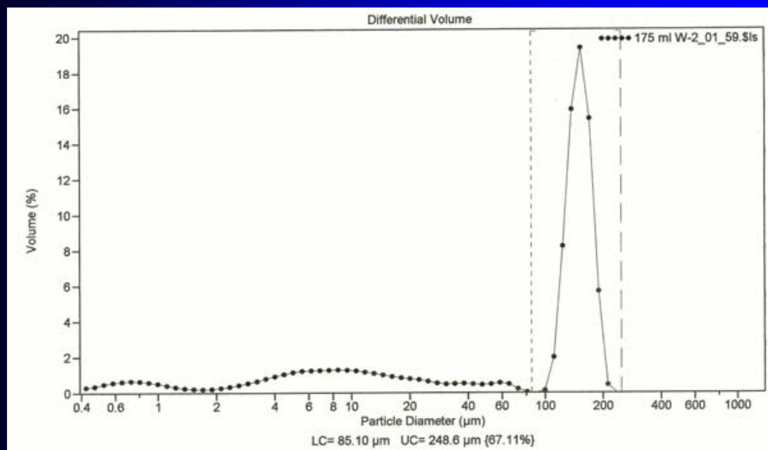
Measure the particle size of dry cement, silica flour, sand etc. Also liquids



Particle size analyzer



Particle size analyzer



Spacer Wettability Apparatus

This apparatus is used to determine the apparent wettability of cement spacer systems, to clean nonaqueous drilling fluids. Cement will not bond to oil-wet surfaces

When the spacer is properly used:

- **Prevent mud / cement contamination**
- **Better bonding potential**
- **Ensure a proper annular seal**

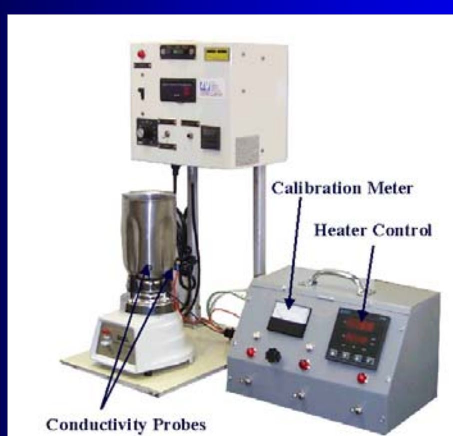
Spacer Wettability Apparatus

Double-walled metal waring blender jar with conductivity sensor, tied to a control box.

•Oil external drilling fluids will not conduct an electric current

•Water-based spacer will As the surface in the jar changes from oil wet to water wet

Wettability Tester



Compatibility Testing

- Spacer / Mud
  - Viscous mixtures
  - Precipitation
- Spacer / Cement
  - Viscous mixtures
  - Premature cement setting
- Mud or Displacement Fluid / Cement
  - Viscous mixtures
  - Premature cement setting

Compatibility Testing (cont.)

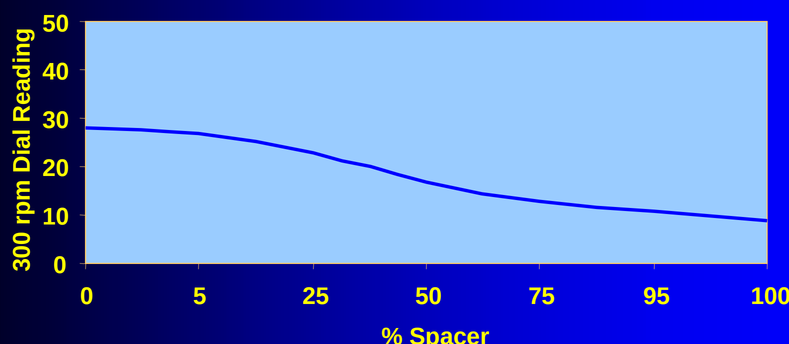
- Take rheology readings of mixtures
  - 100% spacer0% mud
  - 95% spacer 5% mud
  - 75% spacer 25% mud
  - 50% spacer 50% mud
  - 25% spacer 75% mud
  - 5% spacer 95% mud
  - 0% spacer 100% mud

Example Test Results

Mud/Spacer Mixture % by volume	Fann-35 Dial Reading					
	600	300	200	100	6	3
100% Mud	42	28	22	15	4	3
95% Mud / 5% Spacer	40	27	21	14	4	3
75% Mud / 25% Spacer	35	23	19	13	3.5	3
50% Mud / 50% Spacer	25	17	13	9	3	2.5
25% Mud / 75% Spacer	20	13	11	7	2.5	2
5% Mud / 95% Spacer	16	11	9	6	2	1.5
100% Spacer	12	9	7	5	1.5	1

Example Test Results (cont.)

Spacer Rheology	PV	3	n'	0.4150
	YP	6 lbs/100ft^2	K'	0.0145 lb•sec$^{n'}$/ft^2



Example Test Results

Mud/Spacer Mixture % by volume	Fann-35 Dial Reading					
	600	300	200	100	6	3
100% Mud	42	28	22	15	4	3
95% Mud / 5% Spacer	40	27	21	14	4	3
75% Mud / 25% Spacer	35	23	19	13	3.5	3
50% Mud / 50% Spacer	60	32	25	19	18	18
25% Mud / 75% Spacer	20	13	11	7	2.5	2
5% Mud / 95% Spacer	16	11	9	6	2	1.5
100% Spacer	12	9	7	5	1.5	1

Loss Circulation Apparatus

Atmospheric temperature conditions apparatus

Used to test Loss Control Material performance

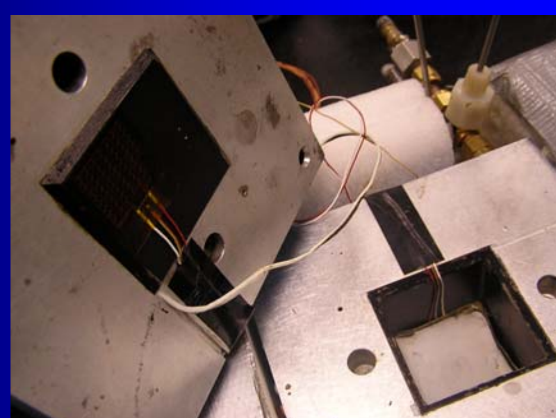
Test done under pressure conditions, with different slots size

Loss Circulation Apparatus

Heat of Hydration Apparatus

Atmospheric conditions apparatus

Used to measure the heat while the cement sets



Advantages of Foam Cement

Over Conventional Lightweight Slurries

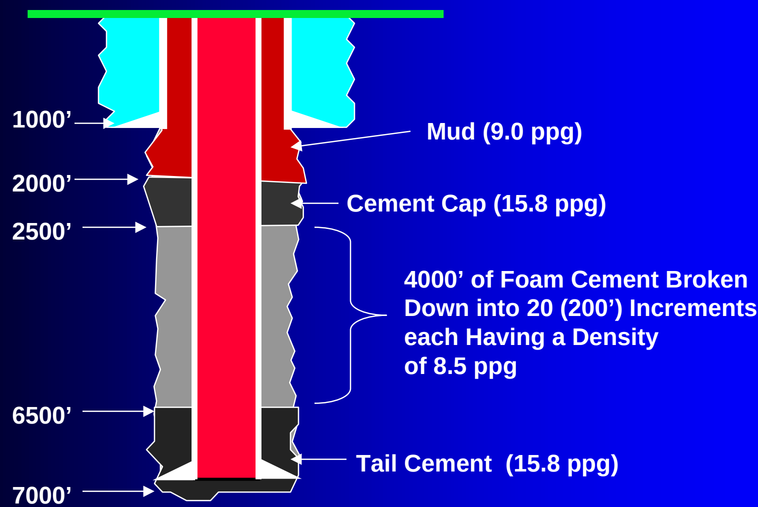
- Densities Less than 8 ppg Possible
- Higher Compressive Strength
- Lower Fluid Loss
- Compatible with All Cement Additives
- Better Thermal Insulating Abilities
- Less Expensive (Microspheres)

Foam Quality

Is the ratio between the volume occupied by the gas and the total volume of the foam, expressed as a percentage.

- Characterizes Foams:
  - Concentrated Foam - Mostly Gas with Thin Film Separating Bubbles (> 80% Quality)
  - Dilute Foam - Spherical Bubbles Separated By Viscous Film (Normally <50% Quality)
    - Foam Cements are in this group - Normally stable cement foam quality is in the 20 to 30% range.

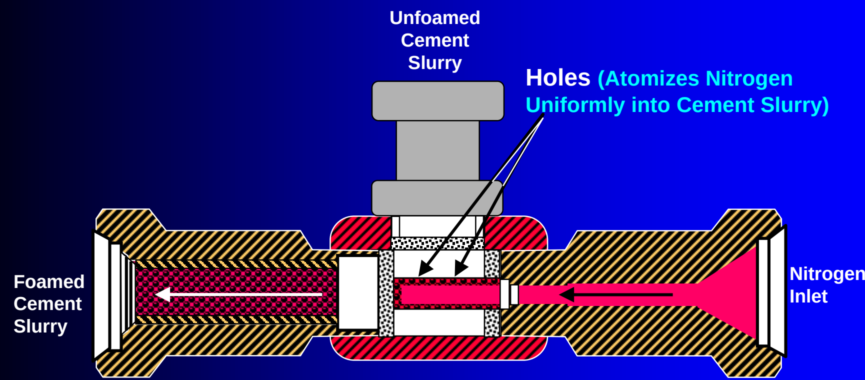
Wellbore Diagram



Example Calculations for One Increment

- HH = 1347 psi
  - 2000’ x 9.0ppg x 0.052 = 936 psi
  - 500’ x 15.8ppg x 0.052 = 410.8 psi
  - 936 + 410.8 =1347 psi
- Temp. = 117°F
  - (2500/100) x 1.5 + 80 = 117°F
- ND = 0.802 ppg
  - Extrapolated from Table 1 (at HH & T)
- PFCD = 15.8 ppg
- FCD = 8.5 ppg
- CY = 1.948
  - (15.8 - 0.802) / (8.5 - 0.802) = 1.948
- NVF = 459 scf/BBL
  - Extrapolated from Table 2 (at HH & T)
- NCR = 435 scf/BBL of Cmt.
  - 459 x (1.948 - 1) = 435

Foam Generator "T"



- Requires 2500 psi Back-Pressure
- Generates Stable, Uniform Foam in 4' of 2.5" Pipe

BJ Tensiometer

HP HT conditions apparatus

Equipment Cure and Test Cement For Tensile Strength Under Down-Hole Conditions (7000 psi/500°F)

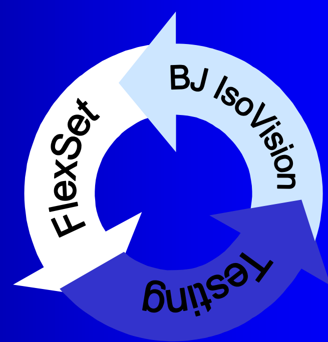
- **Patented Technology**
- **First of it's Kind in the <u>Industry</u>**
- **Three Sample Testing**

BJ Tensiometer

BJ Tensiometer

LOTIS Applications

- **LOng Term ISolation (LOTIS)**
  - **Umbrella of Mechanical Properties Testing Within BJ**
    - **FlexSet Systems**
    - **BJ Tensiometer**
    - **Isovision**



LOTIS Video

END

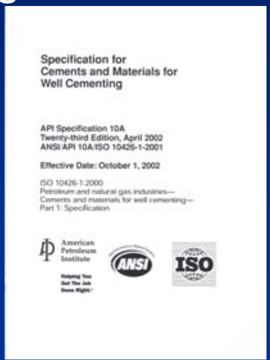
Cement Laboratory Testing

Section 8b

Printed: 4/7/2006

API Testing

- API Specifications and Recommendations
- Definitions
- Temperatures
- Laboratory Testing



Specification for
Cements and Materials for
Well Cementing

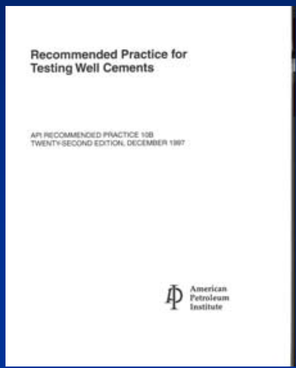
API Specification 10A
Twenty-third Edition, April 2002
ANSI/API 10A/ISO 10426-1-2001

Effective Date: October 1, 2002

ISO 10426-1:2000
Petroleum and natural gas industries—
Cements and materials for well cementing—
Part 1: Specification

American Petroleum Institute

ANSI ISO



Recommended Practice for
Testing Well Cements

API RECOMMENDED PRACTICE 10B
TWENTY-SECOND EDITION, DECEMBER 1997

American Petroleum Institute

API Specifications for Cements and Materials for Well Cementing

- API Spec 10A (October 2002)
  - ISO 10426-1:2000
  - Specifies Chemical, Physical and Performance requirements for API cements and neat slurries.
    - MgO, SO3, C3A, C3S, C4AF
    - Loss on ignition, Insoluble residue, Fineness
    - Compressive Strength, Free Water, Thickening Time
  - Specifies required equipment
    - Calibration procedures
    - Testing procedures

API Recommended Practice for Testing Well Cements

- API RP 10B-2 (July 2005)
  - Specifies procedures for testing and determining properties of cement slurries and set cement.
    - Sampling, Preparation, Density
    - Compressive Strength, Thickening Time, Fluid Loss
    - Permeability, Rheology, Stability, Compatibility
  - Specifies required equipment
    - Calibration procedures
    - Testing procedures

Definitions

- BHST
  - Bottomhole Static Temperature, °F
- BHCT
  - Bottomhole Circulating Temperature, °F
- BHSqT
  - Bottomhole Squeeze Temperature, °F
- BHLT
  - Bottomhole Logging Temperature, °F

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Definitions

- PsTG
  - Pseudo Temperature Gradient, °F/100 ft
  - Similar to geothermal gradient
- MD
  - Measured Depth, ft
- TVD
  - True Vertical Depth, ft

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Temperature

- Most Important factor in slurry design
- Higher temperature
  - Escalates Hydration
  - Decreases thickening time
  - Generally decreases slurry viscosity
  - Generally increases fluid loss, compressive strength, free fluid and segregation/settling

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PsTG

- Pseudo Temperature Gradient, °F/100 ft
  - Similar to geothermal gradient
    - BHST = 80 + PsTG x TVD / 100
      - PsTG = Pseudo Temperature Gradient, °F/100 ft
      - TVD = True Vertical Depth, ft
  - Assumes linear geothermal gradient
  - Assumes surface temperature of 80°F
  - Maps exist for geothermal gradients in US

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BHST

- Bottomhole Static Temperature
  - Can be estimated
    - BHST = 80 + PsTG x TVD / 100
      - PsTG = Pseudo Temperature Gradient, °F/100 ft
      - TVD = True Vertical Depth, ft
  - Measurements are preferred

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BHST from Temperature Logs

- Logging Temperature (BHLT)
  - BHST = BHLT, if the well has been static 36 hours or more
  - If static time is < 36 hrs, use one of two methods to estimate BHST

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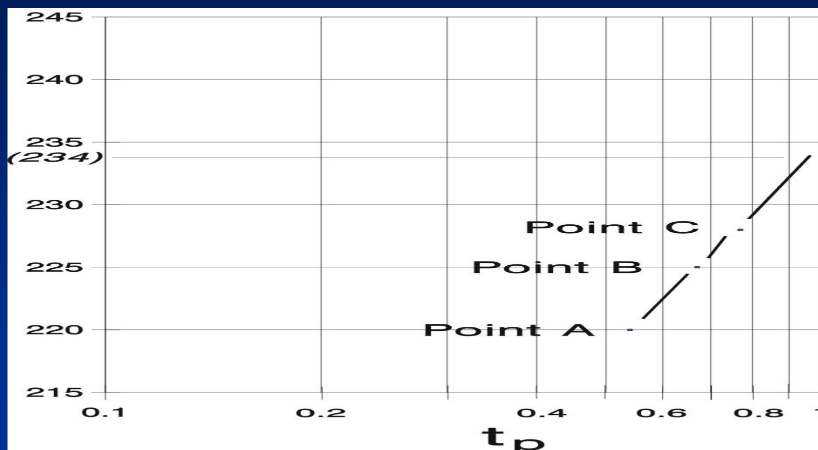
Method 1
(Multiplication Factor)

Time Since Last Circulation	Multiplication Factor (BHST/BHLT)
0 – 6 hours	1.20
6 – 12 hours	1.18
12 – 18 hours	1.15
18 – 24 hours	1.12
> 36 hours	1.00

Method 2
(Extrapolation)

- Need temperature from three log runs
  - t = circulation time, hrs
  - Δt = time after circulation (static), hrs
  - t_p = dimensionless time = $\Delta t/(t + \Delta t)$
- Plot BHLT vs t_p on semi-log scale
- Extrapolate BHST @ $t_p = 1$

Method 2
(Extrapolation)



BHCT

- Bottomhole Circulating Temperature
  - Definition
    - BHCT is the temperature the slurry will obtain while being pumped into place
  - Function of:
    - BHST
    - Circulating rate
    - Circulating time
    - Surface temperature
    - Depth
    - Mud type (oil or water)
    - Hole geometry
    - Rock type

BHCT

- Notes on BHCT
  - BHCT is much lower than BHST
  - Maximum temperature occurs 1/3 to ¼ of the way up the annulus
  - Time-dependent (true steady state is never attained)
    - After 1 or 2 complete circulations, the temperature change is negligible
  - While tripping, wellbore fluid heats up to within 10% of geothermal gradient after 15 hours of trip time
  - API data for casing BHCT was collected from 66 wells
    - API data standard deviation is 16.6 °F

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Determining BHCT

- Casing or Liner Jobs
  - Depths < 10,000 ft
    - BHCT is estimated from API Spec 10, Table 4, Schedules 9.2 – 9.7 and Table 5, Schedules 9.14 – 9.19
  - Depths > 10,000 ft

$$BHCT = 80 + \frac{\left(0.006061 \, x \, TVD \, x \, PsTG\right) - 10.0915}{1.0 - \left(0.1505 \, x \, 10^{-4} \, x \, TVD\right)}$$

    - Standard Deviation = 16.6 °F
  - Or use BJ WellTemp™ Software

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Determining BHCT

- Temperature tables from the Cement Engineering Support Manual

Cementing Engineering Support
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Revision No. 0 Revision Date: 01/01/2001 Printed: 01/25/2005

Section: **6.0 TEMPERATURE INFORMATION**
6.3 Static, Circulating and Squeeze Temperature vs. Depth

TABLE 2
Static, Circulating and Squeeze Temperature versus Depth

Depth TVD FT	0.9°F/100'			1.0°F/100'			1.1°F/100'			1.2°F/100'		
	BHST	BHCT	BHSqT	BHST	BHCT	BHSqT	BHST	BHCT	BHSqT	BHST	BHCT	BHSqT
500	85	80	80	85	80	80	86	80	80	86	80	80
1000	89	80	80	90	80	80	91	80	80	92	80	80
1500	94	84	82	95	84	83	97	84	84	98	85	86
2000	98	89	86	100	89	87	102	89	89	104	90	90
2500	103	91	89	105	92	91	108	92	93	110	93	95
3000	107	94	93	110	95	95	113	95	97	116	95	100
3500	112	96	96	115	97	99	119	97	102	122	98	105
4000	116	99	100	120	100	10	3124	100	106	128	101	109

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Example Calculation BHST/BHCT

- Vertical Well
  - Geothermal Gradient 1.5°F/100ft
  - Depth 8,000ft
  - BHST = 80 + (1.5 x 8,000/100) = 200 °F
  - BHCT = 140°F
    - Cement Engineering Support Manual Section 6.3 Temperature Information, Table 2

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BHCT in Horizontal Wells

- API correlations were developed for vertical wells
  - For Wells with < 30° inclination
    - Estimate BHST normally
- In horizontal wells, slurry is circulated at maximum TVD for a much longer time
  - Use BJ WellTemp™ Software

    or
  - Estimate with Alternative Calculation method

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BHCT in Horizontal Wells (cont.)

- Alternate Calculation Method
  - For Wells with > 30° inclination
    - Calculate PsTG from measured depth (MD) instead of TVD

$$PsTG = \frac{(BHST - 80)}{MD} x 100$$

    - Look up or calculate BHCT using PsTG and MD

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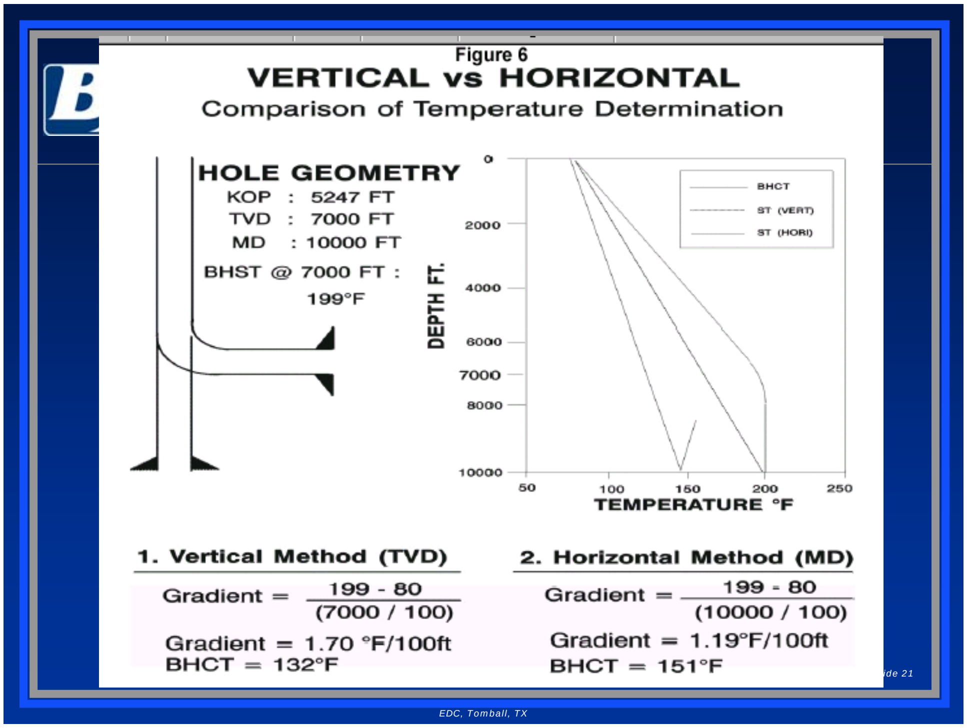

BHSqT

- Bottomhole Squeeze Temperature
  - Squeeze temperatures are generally higher than circulating temperatures
  - BHSqT may be estimated from API Spec 10
    - Table 6, Schedules 9.26 – 9.37 (Continuous Pumping Squeeze)
    - Table 7, Schedules 9.38 – 9.49 (Hesitation Squeeze)

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BHSqT (cont.)

- BHSqT may also be predicted with this formula:

$$BHSqT = 80 + \frac{(0.0076495\ x\ TVD\ x\ PsTG\ ) - 8.2021}{1.0 - (\ 0.807\ x\ 10^{-5}\ x\ TVD\ )}$$

Standard Deviation = 13.0 °F

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Laboratory Testing

- Slurry Preparation & Conditioning
- Thickening Time
- Fluid Loss
- Rheology
- Gel Strength
- Thixotropy
- Compatibility
- Compressive Strength
- Flexural & Tensile Strength
- Free Fluid
- Slurry Segregation/Settling
- Gas Flow Model

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Slurry Preparation and Conditioning

- Mixing
  - Simulates field mixing conditions
  - Mix the water, cement and additives in API mixer (Waring blender) at low speed
    - 4,000 rpm during 15 seconds
  - Shear at high rate
    - 12,000 rpm for 35 seconds

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Slurry Preparation and Conditioning (cont.)

- Conditioning
  - Simulates slurry agitation while traveling through the pipe
  - Place slurry in consistometer and continue stirring while heating up to BHCT and pressuring up to BHP

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API Mixer

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Consistometers

Pressurized Consistometers

Atmospheric Consistometers

Fann 35 Rotational Viscometer

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Consistometer Parts

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Thickening Time

- Pumpability time
  - Measured by Consistometer
    - Atmospheric (BHCT < 194 °F)
    - Pressurized
  - Bearden units of consistency, B_c
    - Related to torque imparted on the paddle shaft
    - Measured with a voltage potentiometer
    - Slurry is usually considered unpumpable at 70 – 100 B_c
  - Test is performed at BHCT
    - Conditioning time, heat-up rate and pressure are determined by API Spec 10 tables
    - Once BHCT is reached, it is maintained constant

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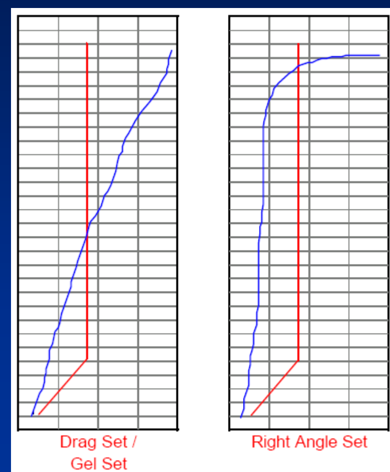
Thickening Time (cont.)

- Types of slurry:
  - Gel Set
    - Drag Set
  - Right Angle Set



Thickening Time (cont.)

- Batch Mixing
  - Slurry is conditioned (stirred) at atmospheric conditions to simulation batch mixing time
    - Typically one hour
  - Reported thickening time does not include batch mix time
- Hesitation Squeeze
  - Second temperature heat-up (ramp) from BHSqT to BHST
  - Slurry stirring is cycled on/off during second temperature ramp to simulate hesitation method
  - Generally gives shorter thickening time than Continuous Pumping Squeeze

Fluid Loss

- Fluid loss is the rate at which water will be forced out of the cement slurry into permeable formations, expressed in mL/30 min
- Measured with a Fluid Loss Cell
  - Slurry is mixed and conditioned to BHCT before doing leak-off
  - Cell consists of a pressurized cylinder with a 325 mesh screen insert to simulate permeable formations
  - 1,000 psi pressure differential is applied
  - Filtrate is collected during a 30 minute interval and measured
  - Standard Cell or Stirred Fluid Loss Cell

Fluid Loss Test at High Temperature

- BHCT > 194 °F
- Method 1 (Standard Fluid Loss Cell)
  - Condition slurry to BHCT in pressurized consistometer
  - Cool slurry to 194 °F
  - Transfer slurry to pre-heated fluid loss cell
  - Increase temperature to BHCT
  - Perform fluid loss test

Fluid Loss Test at High Temperature

- BHCT > 194 °F
- Method 2 (Stirred Fluid Loss Cell)
  - Condition slurry to BHCT in stirred fluid loss cell
  - Invert cell
  - Apply differential pressure
  - Perform fluid loss test
- Safer, Easier & More Representative

Standard Fluid Loss Cells & Parts

Stirred Fluid Loss Cell

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Rheology

- Rheology is the study of flow and deformation of fluids
- Needed to calculate friction pressures and to predict flow regimes
- Rheology is the relationship between flow rate (shear rate) and the pressure (shear stress) needed to move a given fluid
  - Shear Rate (SR) = difference in velocity of two fluid particles divided by the distance between them
  - Shear Stress (SS) = frictional force created by the two fluid particles rubbing against each other

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Fann-35 Rotational Viscometer

- Stationary cup and rotating sleeve
- Internal shaft & bob
- Shear Rate
  - Proportional to rotational speed
  - Shear Rate = 1.7023 X RPM
- Shear Stress
  - Proportional to torque imparted on shaft
  - Shear Stress = 1.065 x Dial Reading

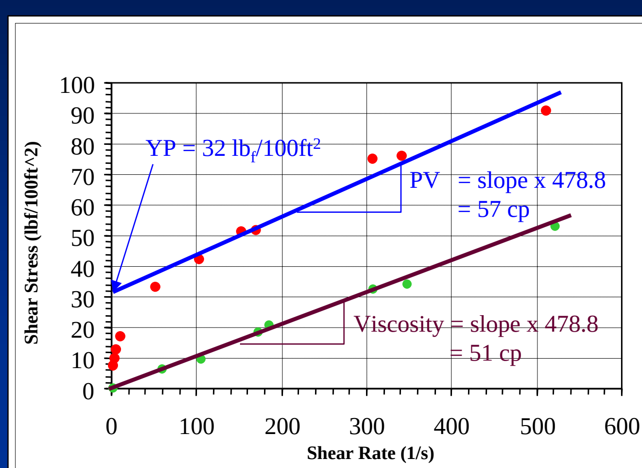
Fann-35 Rotational Viscometer (cont.)

- Measurements are generally taken at ambient temperature (to simulate mixing conditions) and BHCT (to simulate pumping conditions)
- General Rules of Thumb
  - Low end readings (3 & 6 rpm) of less than 5 indicate the possibility of solids settling
  - A low end reading of greater than 20 indicates a strong possibility of gelation
  - High end readings (300 & 600 rpm) of greater than 200 could indicate difficulty in field mixing and pumping

Rheological Models

- Newtonian Model
  - SS is directly proportional to SR
  - Viscosity = slope of SS vs SR x 478.8, cp
  - Water, gasoline, diesel, light oils
- Bingham Plastic Model
  - Fluid will remain static until a certain minimum SS is applied, then SS is proportional to SR
  - Yield Point (YP) = minimum SS to move fluid, $lb_f/100\ ft^2$
  - Plastic Viscosity (PV) = slope of SS vs SR x 478.8, cp
  - Cement slurries, drilling muds, cementing spacers and preflushes

Newtonian vs Bingham Plastic



Rheological Models (cont.)

- Power Law Model
  - SS is proportional to SR to the power of n’
    - Log(SS) is proportional to log(SR)
  - Flow Behavior Index (n’)
    - Slope of log(SS) vs log(SR)
    - Normally, n’ is less than one
  - Consistency Index (K’)
    - Intercept of log(SS) vs log(SR) / 100 * ((3n’ + 1)/4n’)^n’
    - Units in $lb_f \cdot s^{n'}/ft^2$
  - Cement slurries, drilling muds, cement spacers and preflushes

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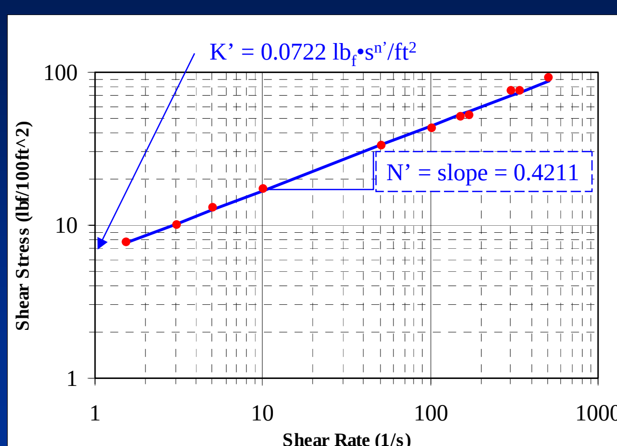
Power Law Model



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Choosing Rheological Model

- Plot Shear Stress vs Shear Rate on linear and log-log coordinates
- Determine which coordinates give the best straight line:
  - If linear: Use Bingham Plastic
  - If log-log: Use Power Law
- Alternatively, use linear regression technique to determine coefficient of regression
  - Choose model with coefficient of regression closest to 1.000

Gel Strength

- Force required to initiate fluid movement
- Measure on Fann-35
  - Mix and condition slurry to BHCT
  - Take rheology readings
  - Stir for 60 seconds at 600 rpm
  - Stop for 10 seconds
  - Turn on 3 rpm, observe maximum dial reading (will break back),multiply by 1.065
    - 10 second gel strength
  - Stop for 10 minutes
  - Repeat 3 rpm reading, multiply by 1.065
    - 10 minute gel strength

Gel Strength (cont.)

- Pressure required to start movement of a column of fluid is a function of the gel strength, column height, and cross-sectional area

$$P = Sg \times L / (300 \times D)$$

- P = Pressure, psi
- Sg = Gel Strength, lbs/100 ft2
- L = Length, ft
- D = Casing Diameter, inches

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Thixotropy

- Thixotropic slurries become semi-solid at rest and liquid when agitated
- Test with Fann-35
  - After standard gel test, agitate at 600 rpm for 60 seconds, stop 10 seconds, measure initial gel strength, G(i)
  - Leave slurry static for 10 minutes
  - Agitate at 600 rpm for 60 seconds, stop 10 seconds, and measure gel strength again, G(f)
  - To be considered thixotropic, G(f) should be at least 20% greater than G(i)

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Thixotropy (cont.)

- Alternate Test Method (Cup)
  - Pour slurry in paper or styrofoam cup
  - Leave static 2 minutes
  - Invert cup to test for pourability
  - Repeat every minute until slurry is unpourable
  - Stir with glass rod 15 seconds
  - Check if slurry is pourable, if yes, then record time as initial gel set time
  - Repeat procedure
  - A good slurry should be able to be sheared 3 times before becoming too viscous to pour

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Compatibility Testing

- Spacer / Mud
  - Viscous mixtures
  - Precipitation
- Spacer / Cement
  - Viscous mixtures
  - Premature cement setting
- Mud or Displacement Fluid / Cement
  - Viscous mixtures
  - Premature cement setting

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Compatibility Testing (cont.)

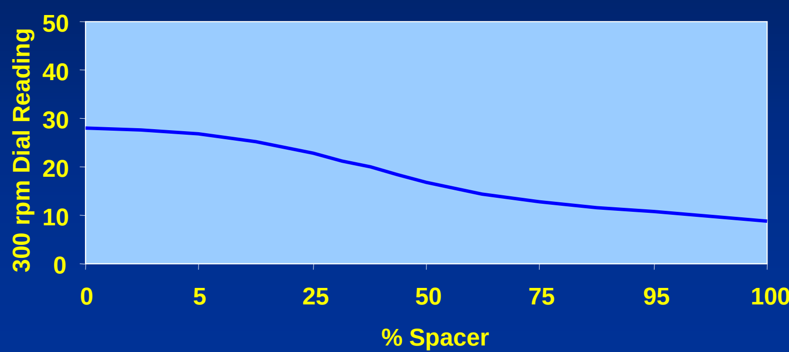
- Take rheology readings of mixtures
  - 100% spacer 0% mud
  - 75% spacer 25% mud
  - 50% spacer 50% mud
  - 25% spacer 75% mud
  - 0% spacer 100% mud

Example Test Results

Mud/Spacer Mixture % by volume	Fann-35 Dial Reading					
	600	300	200	100	6	3
100% Mud	42	28	22	15	4	3
95% Mud / 5% Spacer	40	27	21	14	4	3
75% Mud / 25% Spacer	35	23	19	13	3.5	3
50% Mud / 50% Spacer	25	17	13	9	3	2.5
25% Mud / 75% Spacer	20	13	11	7	2.5	2
5% Mud / 95% Spacer	16	11	9	6	2	1.5
100% Spacer	12	9	7	5	1.5	1

Example Test Results (cont.)

Spacer Rheology	PV	3	n'	0.4150
	YP	6 lbs/100ft^2	K'	0.0145 lb•sec$^{n'}$/ft^2



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Wettability Tester

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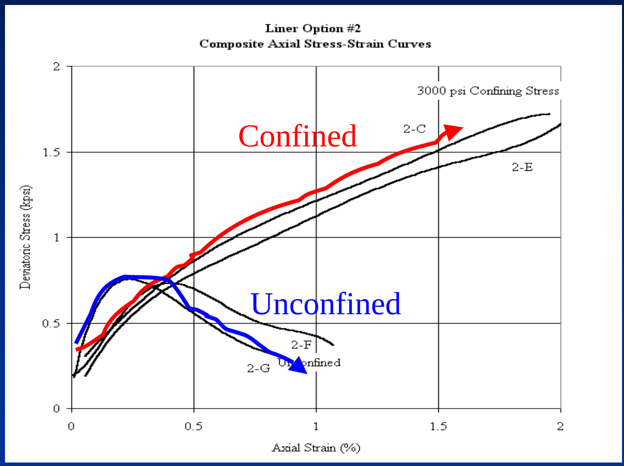
Compressive Strength

- Measured in psi, and is a function of Time and Temperature
- How much do we need?
  - Traditional rules of thumb
    - 5 to 200 psi to support casing
    - 500 psi to continue drilling
    - 1,000 psi to perforate
    - At least 2,000 psi to stimulate & isolate zones
    - Enough strength to side track (more than adjacent formation)
  - Mechanical integrity calculations and experience show we may not need as much as we traditionally thought
    - LOTIS Technology and IsoVision software

Compressive Strength
Effect of Confining Stress

	Unconfined Measurements UCS psi	Confined Measurements C.S. psi	Confining Stress psi
Class G	7160	11,720	3,000
	6,040	15,130	5,000
	3,520	11,800	5,000
	4,700	12,150	5,000
Neat	3,540	9,200	1,000
		12,500	2,000
		14,500	4,000
Class H	1,780	13,700	3,000
	765	8,410	3,000
	10,500		
	4,280		

Compressive Strength
Effect of Confining Stress



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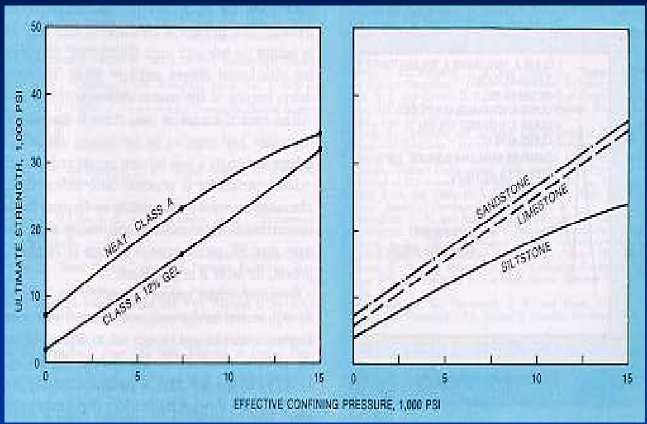
Ultimate Confined Compressive Strength



Source – World Oil 1977

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Measured Compressive Strength

- Unconfined Compressive Strength
- Destructive Test
  - Prepare conditioned slurry in 2 in^2 cube molds
  - Cure in curing chamber to BHST
  - Load to failure in hydraulic press at different elapsed times
  - UCS = Force / Area (psi)

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Measuring Compressive Strength

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Ultrasonic Cement Analyzer

- Non-destructive test
- Measures and records the inverse P-wave of velocity through a cement slurry as a function of time
- Unconfined compressive strength is estimated via an empirical algorithm
- Continuous read-out
- Also plots sonic travel time, in order to calculate attenuation time to calibrate cement bond logs

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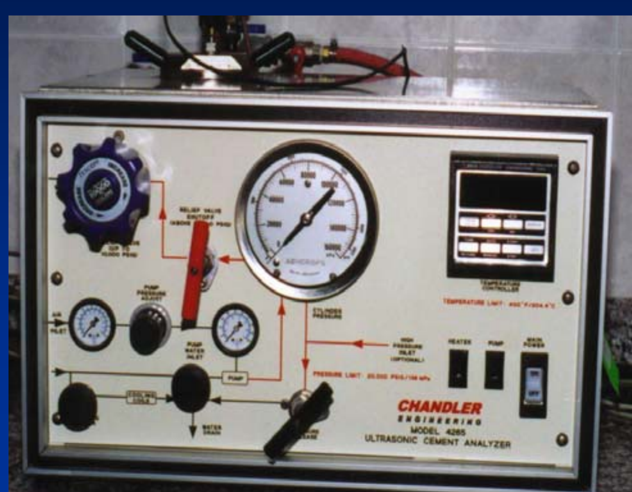
Ultrasonic Cement Analyzer (cont.)



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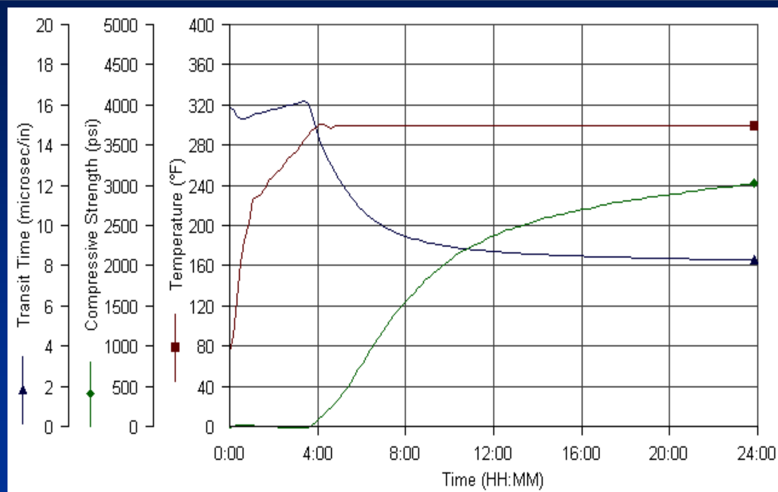
Ultrasonic Cement Analyzer (cont.)



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Flexural & Tensile Strength

- **Cement slurry prepared and cured in special molds**
- **Flexural Strength measured using the lever and fulcrum effect**
- **Tensile strength measured by applying tension to shaped mold**
- **Recent evidence shows that tensile strength may be more important than compressive strength**
  - **Ductility, flexibility**
  - **Stress cycling**

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Flexural & Tensile Strength (cont.)

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Flexural & Tensile Strength (cont.)

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Flexural & Tensile Strength (cont.)

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Free Fluid

- Free fluid may separate out and contribute to slurry shrinkage
- Shrinkage may affect bonding or contribute to gas migration
- In deviated wellbores, free fluid will float to the high side of the wellbore and create a conductive channel

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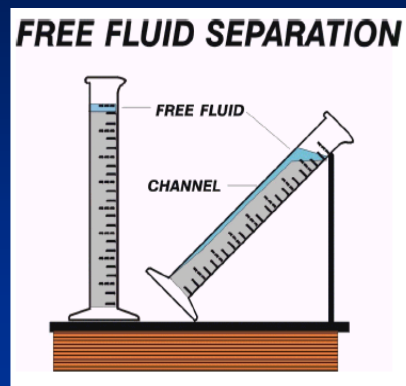
Free Fluid Test

- Mix and condition slurry to BHCT
- Pour slurry into 250 mL graduated cylinder
- Leave static 2 hours at ambient temperature (may be inclined to simulate wellbore)
- Measure free fluid with a pipette



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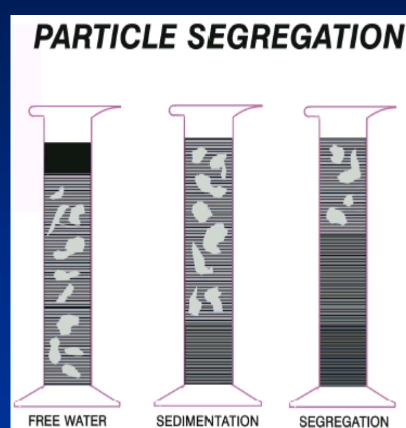
Slurry Segregation & Settling

- Test measures the ability of the slurry to maintain a stable suspension at downhole conditions
- Critical for deviated and horizontal wellbores and for gas-migration control slurries



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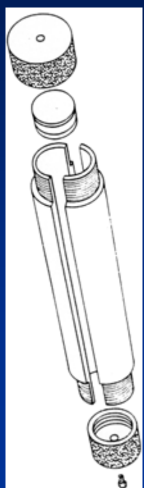
BP Settling Test

- Mix and condition slurry to BHCT or 194 °F
- Transfer to preheated settlement tube
- Place in curing chamber, ramp to BHCT and maintain until end of test, applying 3,000 psi
- After 16 hours, cool to ambient temperature
- Measure settlement, in mm
- Break column into 1 inch segments
- Measure density of each segment by Archimedes method

BP Settling Test



Gas Migration Testing

- Simulates exposure of cement slurry to a high-pressure permeable gas zone and to a lower pressure permeability zone
- The hydrostatic pressure of the fluid on top of the cement plus the hydrostatic pressure of the unset cement will prevent gas intrusion from occurring
- During the cement hydration process, the hydrostatic pressure on top of the slurry is relived thus reducing the cement pore pressure
- As the cement sets, the cement pore pressure may decrease below the gas reservoir pressure. The unbalance of pressures can allow gas to intrude the cement column.
- The gas can migrate to the well and to the surface and may cause a well blow out or the gas can communicate to a lower pressure permeable zone

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Gas Flow Model

- Cement slurry is mixed and conditioned to BHCT
- Three pressures are applied to the cement slurry during the test
  - Pressurized mineral oil will simulate the hydrostatic pressure of different fluids like drilling mud or cement spacer
  - The gas pressure is applied into cement slurry with nitrogen gas
  - The third pressure is applied with the use of a back pressure regulator to simulate a low-pressure permeable zone
- Piston movement, fluid filtrate volume and gas filtrate volume and cement pore pressure are measured continuously during the test

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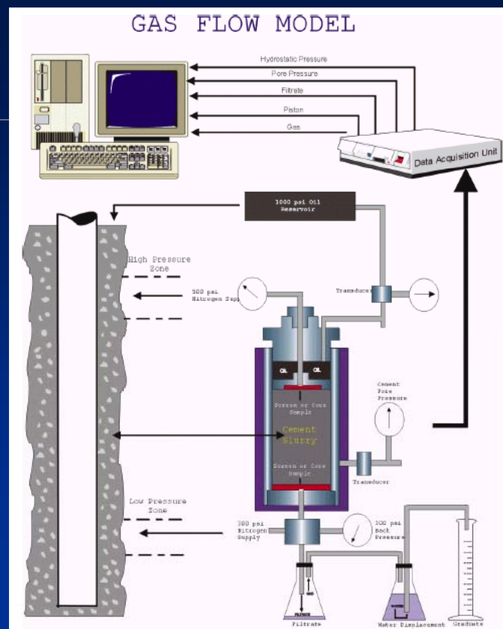

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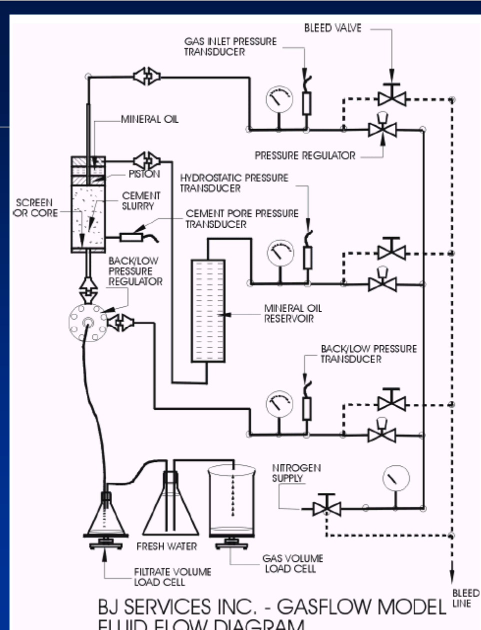

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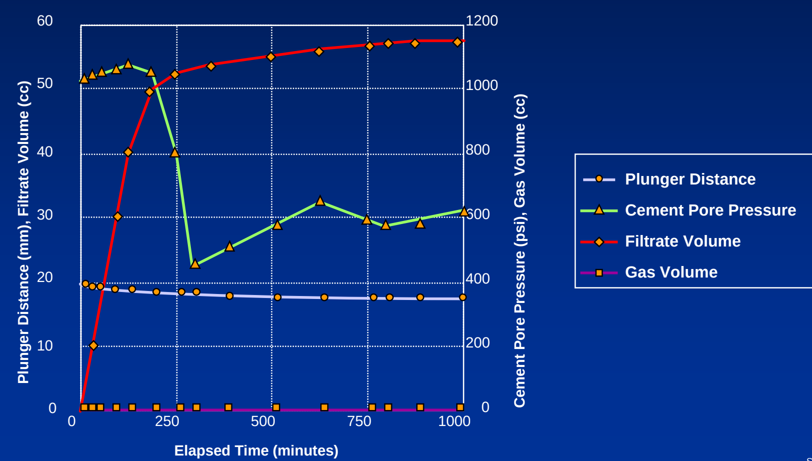
Typical Gas Flow Model Results



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Density Measurement

Atmospheric Mud Scale

Pressurized Mud Scale

EDC, Tomball, TX

Shear Bond Test

Measures hydraulic coupling to steel

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EDC, Tomball, TX

Cement Expansion / Shrinkage

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EDC, Tomball, TX

Cement Expansion / Shrinkage (cont.)

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EDC, Tomball, TX

References

- Cementing Engineering Support Manual
  - CementEngSupport.nsf
- API Spec 10A
  - Cements and Materials for Well Testing
- API RP 10B
  - Testing Well Cements

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EDC, Tomball, TX